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THE MARTENSITIC TRANSFORMATION IN IRON - NICKEL ALLOYS

by

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A Thesis

Submitted to the Faculty of Graduate Studies in Partial Fulfilment of the Requirements for the Degree of Master of Science.

Department of Mining and Metallurgy

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ABSTRACT

A quenching unit in which small specimens may be heated to 1,000 °C at a pressure of 10^{-5} mm. Hg, and quenched with a blast of high purity helium is described. Quenching rates as high as 55,000 °C/sec. are reported.

The M_s temperatures for alloys of iron with 1, 2, 3, 5, 7, and 10 at. pct. nickel have been determined, and related studies of pure iron are also reported. The M_s temperatures decrease progressively with increasing nickel content. The martensitic transformation takes place only when a critical cooling rate has been reached, the value of which decreases with increasing nickel content.

The results of the present work agree with some of the earlier published results, disagree with most, and provide new information about the martensitic transformation in low nickel - iron alloys.

ACKNOWLEDGEMENTS

The author is very grateful to Dr. J. G. Parr for his valuable advice in this project. The encouragement of Professor E. O. Lilge has greatly facilitated this work.

He also wishes to express his appreciation to Malcolm J. Bibby for his assistance and suggestions, and to Mr. R. M. Scott for his very competent assistance with photographic problems.

Particular gratitude is extended to the Defence Research Board of Canada, whose financial support made this work possible.

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INTRODUCTION

The term martensite was first used, in honour of Professor A. Martens (1850 - 1914), to define a constituent in hardened steel.[1] However, Scheil[2] (1929) referred to the formation of martensite in zirconium, but it is only within more recent years that the term has become universally used to describe the product of a certain type of transformation process. Precise definitions will be given later, but for the purposes of introduction, martensite may be considered to be the product of a diffusionless transformation, involving the shear of atomic planes, to produce a new crystallographic arrangement.

Frequently, the martensitic transformation can only be induced by very rapid cooling, which is necessary to prevent the formation of the low temperature equilibrium phase that would otherwise form by a diffusion process and, perhaps, to initiate the martensitic mechanism. Consequently, the attainment of very high cooling rates may be necessary for the production of martensite in some systems ---- of which the iron-nickel system appears to be one. Previous investigations of iron-nickel martensites have been limited to cooling rates about 10,000 °C/sec; but Owen[3] pointed out recently that the transformations previously regarded as martensitic were not, in fact, martensitic. Hence a large amount of data relating the martensitic process in iron-nickel alloys to such parameters as composition and thermodynamic functions have been thrown into doubt.

The general purpose of the work described in this thesis was to apply a generally accepted criterion of martensite formation (that is the "shape change" associated with the process), and to determine under what circumstances martensite was actually produced in alloys of iron with up to 10 at. pct. nickel. This objective necessitated the development of techniques that permitted extremely rapid quenching rates (up to 60,000 °C/sec. have been attained). A comparison of the results with previous work throws into doubt the quantitative aspects of previous theories and, indeed, contributes to a belief that the theories may be wrong in principle.

LITERATURE REVIEW

A. The martensitic transformation in metals and alloys

For the purpose of this review the earliest theories (beginning perhaps with Reaumur's contention in 1722 that quenched steel was hardened because the grains were better held together) can be omitted, since the time-proven hypotheses are incorporated in present-day ideas.

In their review of martensitic transformations, Bilby and Christian[4] define a martensitic transformation in the following way: "A structural transformation is classed as martensitic if the atoms on a primitive lattice defined by a selected unit cell of the parent structure move to positions on a primitive lattice defined by some unit cell of the product structure, in such a way that the displacements constitute a homogeneous deformation, this deformation may be different in adjacent small regions".

The word "martensitic" is hence used to describe a type of transformation rather than the character of a transformation product.

Martensite is a metastable state[5] believed to be formed by a shearing process involving very little atomic movement (of less than one atomic distance) as opposed to transformation by nucleation and growth[6] by diffusion. Cottrell[7] defines a shear process as one in which atoms

move along a shear plane from one set of equilibrium conditions to a new set of equilibrium conditions. After reaching the new position; they do not slip back to their old one when the shear force is removed.

The criterion of "shape change", as proposed by Bilby and Christian[4], is accepted in this thesis; namely, that a martensitic reaction is one involving a shear tilt on a plane surface that lies at an angle to the plane of shear. This implies that the smooth surface of a polished sample of metal or alloy will take on a "rumpled" appearance while undergoing a martensitic transformation. Therefore a transformed sample is not considered to be martensitic unless there is evidence of surface "shape change".

Greninger and Troiano[8], when working on steel containing 0.8 pct. carbon and 22 pct. nickel, proposed that simple shear takes place on the martensite habit plane. They later proposed that two shear processes take place: the first stage consisting of a homogeneous shear on the habit plane producing relief effects, the second stage being a different shear, homogeneous within lamellae so narrow (about 18 atomic planes) that they are invisible, but macroscopically heterogeneous. The second shear occurs on the martensite twinning elements; this together with the fact that martensite plates sometimes show transverse markings parallel to $\{112\}_M$* planes makes the proposal seem reasonable. This mechanism accounts for the relief effects, the habit plane (the plane of the first shear), the orientation relationship (to a good

---

* M ---- means Martensite

approximation), and the change in structure. However, an anisotropic expansion must be assumed in addition to the shears. This amounts to 4.2 pct. in the $[100]_M$ direction. The mechanism also fails to account for the orientation relationship in plain carbon steels containing less than 1.4 pct. carbon, where the habit plane is $\{225\}_A$*[9].

Jaswon and Wheeler[10] proposed a different interpretation of the habit plane: If an atomic plane in the austenite were to be converted into an atomic plane in martensite and were to serve as the habit plane of the martensite plate, it would involve large scale plastic distortion of the surrounding austenite unless this conversion left the plane unrotated, i.e. left the direction of its normal unchanged. Thus they identified the habit plane with one of the crystallographic planes that undergoes no rotation during the transformation.

Martensite starts to form only when austenite is cooled below a critical temperature M_S ("the martensite start temperature") which is dependent upon composition. Increased solute content has the effect of lowering the M_S temperature. While most martensitic reactions do display athermal** characteristics, starting at a well-defined temperature (even during drastic quenching) and continuing only while the temperature is falling, isothermal martensitic transformations have been discovered in several cases, both with and without prior athermal transformation[1].

---

* A ---- means Austenite

** ------ ie. independent of cooling rate

Bowles and Barrett[11] state that many of the phenomena which characterize martensitic transformations seem to indicate that martensite plates develop from nuclei. For example, when a martensitic transformation is reversible, the plates that appear in a certain region during cooling reappear in the same form, in exactly the same region, during a second cooling cycle[12]. It has been concluded from this that embryos exist in the parent phase that nucleate the reaction, and that the sites of these embryos are not necessarily changed on reheating[13]. Fisher, Hollomon and Turnbull[14] suggest that the martensitic transformation occurs by coherent growth of nuclei formed at higher temperatures and have become supercritical in size because of the lowered temperature. They suggest that growth is accomplished by means of thermally activated atom movements, the activation energy being very small, thus permitting growth to occur with extreme rapidity.

The distinctive features of martensitic transformations may be stated in point form as follows:

(1) The martensitic transformation is diffusionless and does not involve a change in chemical composition (i.e. the transformation product inherits the composition of the parent phase)[15].

(2) The transformation occurs by a shear process parallel to the interface (or habit plane) which results in a change in shape of the surface of the metal[4].

(3) The interface remains invariant, i.e. unrotated and undistorted when averaged over macroscopic distances; however,

there is a dilational component normal to it. The interface may be considered to comprise dislocation arrays[16].

(4) The martensitic transformation proceeds by the formation of new lenticular or ellipsoidal plates rather than by growth of pre-existing plates[1].

(5) Once nucleated, each martensite plate propagates quickly (in the order of 1/3 the speed of sound) until it collides with a structural barrier or its mechanism becomes jammed[17].

(6) After the martensitic reaction gets underway the plates being formed begin to diminish in size due to the partitioning of the matrix by the increasing number of plates[1].

(7) Martensitic reactions can propagate at temperatures approaching absolute zero (demonstrated by Kulin and Cohen[18] for iron - nickel and iron - nickel - carbon alloys); the mobility of the interface does not seem impaired by the obvious lack of thermal activation.

(8) The transformation may occur smoothly, or in sudden "bursts" in which a large amount of martensite is suddenly formed and the reaction completed in a fraction of a second[19].

(9) Plastic deformation has the effect of raising the M_s value. Cold-work tends to suppress the transformation when a cold-worked specimen is subsequently cooled[11]. Applied stress will change the M_s temperature[20][21].

(10) The M_s temperature is uniquely determined by the chemical composition of the material and is usually lowered by increasing the solute content in an alloy[5].

(11) Martensite is a metastable phase between the parent phase and the final equilibrium phase, although it may persist for an indefinite period of time at room temperature[5].

(12) Both isothermal and athermal martensitic transformations have been observed. Athermal - kinetic behaviour is shown by iron - nickel[22], and gold - cadmium[23] alloys. Isothermal - kinetic behaviour is shown by iron - nickel - manganese[24] and uranium - chromium alloys[25].

(13) Higher hardness is observed in martensitically transformed steels and many other alloy systems. However, it is not an essential characteristic of a martensitic transformation[5].

B. The iron - nickel transformation.

Iron - nickel alloys transform to martensite on cooling by the successive formation of new plates, with each one shooting out suddenly to its final size[1]. Consequently, the course of the transformation as a function of falling temperature is dependent upon the rate of nucleation and the size of the fully grown plates, but not upon their rate of growth. Such reactions are typified by a high degree of supercooling and have a relatively large driving force below the martensite - start temperature (M_S). Once nucleated, each martensite unit propagates quickly until it collides with a structural barrier or its mechanism becomes jammed. On continued cooling there is no additional

growth of the plate despite the increased driving force; further nucleation occurs elsewhere in the parent phase[1]. In contrast to the transformation in iron - nickel alloys is the gold - cadmium type transformation[1] in which the alloys generate martensite plates that do not reach their final size at once. Instead, although each unit seems to nucleate suddenly and "pop out" to a detectable size within a small fraction of a second, further propagation in length and thickness continues (frequently in jerky fashion) with the decreasing temperature until collision or jamming takes place. Martensitic reactions in this category start with comparatively little supercooling and the driving force is insufficient to supply the strain - energy and other nonchemical requirements of fully grown plates[1]. The martensitic transformation in iron - nickel alloys is considered to be of an athermal nature[1]. However, Yeo[26] has shown that decarburization of an iron - nickel alloy allows the isothermal transformation to martensite to occur at temperatures above their athermal M_s temperature. He shows that in an iron - 22.4 wt. pct. nickel alloy, with 0.008 wt. pct. carbon, the M_s temperature during air cooling is 85 °F (47 °C) higher than that during water quenching. At 0.15 pct. carbon, this difference is reduced to only 5 °F (3 °C).

The introduction of carbon causes stabilization* during air cooling, which lowers the M_s temperature virtually to the same level as determined during water quenching. Thus in the 0.15 pct. carbon alloy, the M_s temperature is almost independent of cooling rate (athermal process).

At this juncture it seems appropriate to point out that the iron - nickel studies for this thesis suggest an athermal martensitic transformation. The M_s temperature is not depressed with increased quench rates. Keeping in mind the work of Yeo[26], it might be suggested that the athermal nature of the transformation may be attributable to the stabilizing effect of the alloy content.

The following treatment has been devised to explain athermal transformations. Knapp and Dehlinger[28] extended Frank's[16] analysis by regarding the embryo of martensite as a thin oblate spheroid, surrounded by loops of dislocations. These loops are assumed to consist primarily of screw dislocations, with short edge components joining the positive and negative screws. Growth of the embryo in the $[1\bar{1}0]_\gamma$ and $[225]_\gamma$ directions is achieved by expansion of the dislocation loops. However, growth in the $[55\bar{4}]_\gamma$ direction requires the generation of new loops. The co-operative movement of these loops leads to the thickening and radial propagation of

---

*Stabilization is a process that reduces the extent of the austenite-martensite transformation. In its most common form it causes an inhibition of the subsequent athermal transformation when cooling is resumed after a temporary arrest. This aspect has been reviewed by Cohen[27].

the martensite embryo as the dislocation interface passes through the austenite[1].

Martensitic reactions can propagate at temperatures approaching absolute zero, as demonstrated by Kulin and Cohen[18] for iron - nickel and iron - nickel - carbon alloys. Evidently, the interface motion is a coordinated or wave-like phenomenon, not involving atom - by - atom transfer across the interface, and neither short range nor long range diffusion is required for the process. The actual movement of each atom relative to its neighbours is very much less than one lattice spacing[1].

Bunshah and Mehl[17] have found that the propagation rate of martensite plates in an iron - 29.5 wt. pct. nickel alloy is about 10^5 cm./sec., with a time of formation of 0.05 - 0.5 μ sec. This corresponds to one third the velocity of sound. Of equal significance is their observation that the propagation of isothermally formed martensite is the same as for the athermal product, and is virtually independent of the temperature over the range studied (-20 to -195 °C). The sound velocity is likewise insensitive to temperature in this alloy.

When the martensitic transformation takes place in an iron - nickel alloy, the crystallographic form changes from the F.C.C. austenite structure to the B.C.C. martensite structure. The (259) plane is (approximately) the habit plane in the parent phase on which shear takes place[11].

EXPERIMENTAL

A. Materials

(1) alloy preparation

Alloys of iron* - 1,2,3,4,5,7,10,15,20, and 25 at. pct. nickel* were prepared in a copper hearth electric arc furnace under a flowing atmosphere of argon at a pressure slightly above atmospheric. Each alloy was melted twice to ensure homogeneity.

Figures 1 and 2 are photographs of the apparatus used. The arc furnace was powered by a 32 volt D.C. arc welder power supply delivering about 160 amperes**, at reverse polarity***. The initial arc was stabilized by superimposing a high frequency source on the power delivered to the electrode. Once a stable arc was established, the high frequency source was shut off.

Metallographic sections of various ingots were prepared to determine homogeneity and grain size. Figures 3, 4 and 5 are typical photomicrographs of these sections. Samples for wet chemical analyses were cut from various areas of the ingots. Results of these analyses indicated

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*See Appendix II for analyses of the materials used, and alloys prepared.
**This current delivered with a welder setting of "medium" and #7 on the relative scale.
***See Appendix I for circuit diagram.

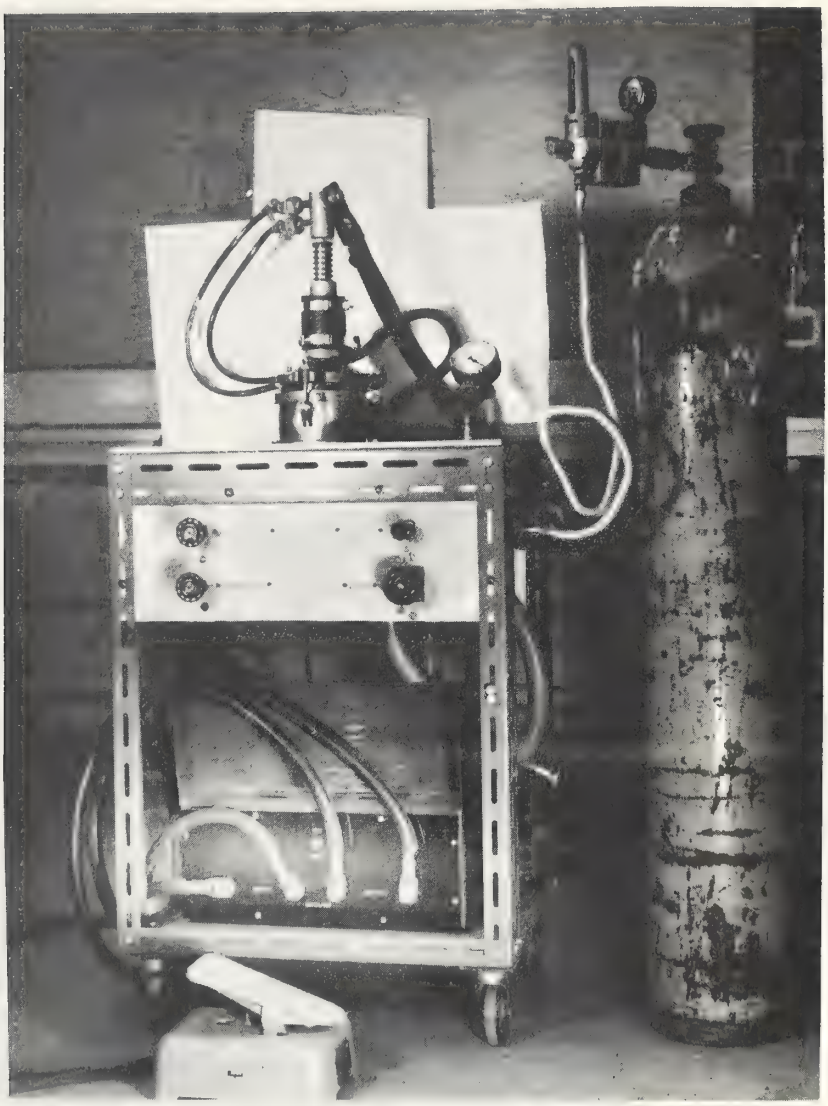

FIG. I. ELECTRIC ARC FURNACE ASSEMBLY WITH FOOT CONTROL, HIGH FREQUENCY ARC STABILIZER, AND ARGON GAS SUPPLY.

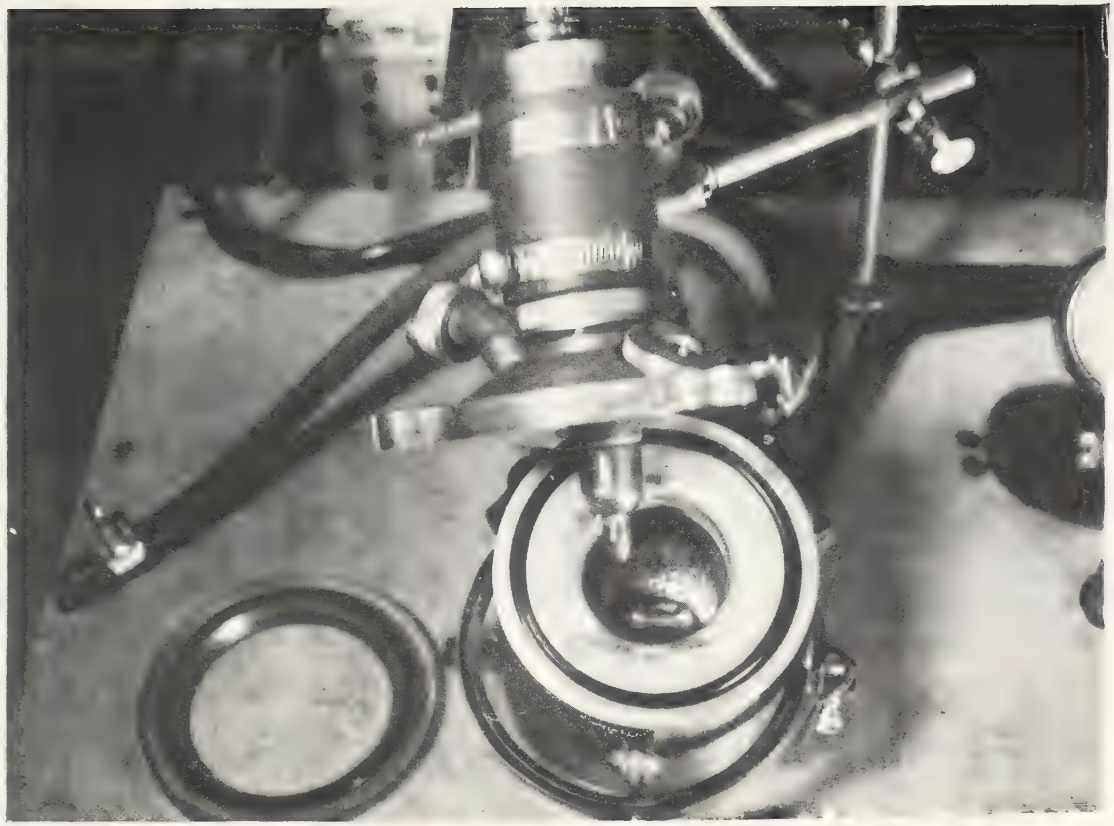

FIG. 2. DISMANTLED VIEW OF ELECTRIC ARC FURNACE, SHOWING THE COPPER HEARTH [WITH SAMPLE IN PLACE] AND THE WATER - COOLED TUNGSTEN ELECTRODE.

homogeneity of the alloy ingots. In addition to the wet chemical analyses, samples were sent out for spectrographic analysis* and were found to be free of contamination. From the results of these tests, the alloy preparation was deemed satisfactory for our purposes.

Each of the alloys displayed a relatively large grain size and was subsequently cold - worked to 75 pct. reduction prior to annealing. Ingots were wrapped in molybdenum foil, sealed in quartz tubes (evacuated to less than 10^{-5} mm. Hg.) and soaked at 935 °C (which is in the austenitic range) for a period of six hours and then quenched in cold water.

This heat treatment produced fine - grained ingots satisfactory for use in the experimental work.

(2) specimen preparation

Specimen preparation consisted of folding a thin slice of the alloy around a chromel - alumel thermocouple (wires of 0.0031 inch diameter) and spot - welding to ensure good electrical contact and high mechanical strength. The rough specimen was then reduced to a size about 0.010 inch square by 0.005 inch thick by filing with a tungsten-point file.

---

*See Appendix II for analyses.

FIG. 3.

FE – 20 AT. PCT. NI., X5, CROSS – SECTION OF INGOT.

FIG. 4.

FE – 20 AT. PCT. NI., X65, SHOWING ALLOY STRUCTURE AND GRAIN BOUNDARIES.

FIG. 5.

FE – 20 AT. PCT. NI., X150, SHOWING WIDMANSTATTEN STRUCTURE AND GRAIN BOUNDARIES.

Accuracy of the thermocouple (after spot - welding the specimen) was checked over a wide range of temperatures, by comparison with values recorded with a chromel - alumel pair (0.040 inch diameter), and was found to be satisfactory ($\pm$ 2 °C).

B. Equipment

The apparatus used in this project consisted of three main sections. These are shown in Fig. 6 and comprised:

(1) quenching unit

(2) vacuum system

(3) data - recording equipment

(1) quenching unit

The finished specimen was placed in the gas - quenching apparatus (Figures 7, 8 and 9).

Fig. 7 is a schematic drawing of the quenching apparatus which comprised a tungsten wire (0.020 inch diam.) heater (3/16 inch diam.) surrounding a small sample supported on its thermocouple leads. A convergent - divergent nozzle (Fig. 10), designed to give supersonic velocities, was connected through a 1/2 inch "Asco" solenoid valve (115 volts, 60 c.p.s., 10.5 watts, with an operating range of zero to 200 p.s.i. air or gas) to a 15 psig. helium or argon supply.

To prevent the sample from being blown out of the quench - gas stream, the thermocouple leads were supported by a quartz member which was, in turn, held in place by a metal clamp fixed to an adjacent pillar.

The nozzle was shielded from the furnace coil by an inverted ceramic crucible with a hole cut in its base; the entire assembly was surrounded by a bell jar that could be evacuated to a pressure of 10^{-5} mm. Hg.

The furnace coil was energised by a 300 VA, 110 V primary, 0 - 15 V secondary, transformer, which in turn was in circuit with a variable voltage supply.

Slow rates of cooling were obtained by allowing the specimen to cool in vacuo. Argon was used to obtain rates to about 12,000 °C/sec. Rates to 60,000 °C/sec. were attained by quenching with high purity helium (99.999 pct.). In addition to the type of quench gas used, sample sizes could also be varied to change the limits of cooling rates.

(2) vacuum system

Each experiment was completed in a vacuum of 10^{-5} mm. Hg. or lower. The system was evacuated by a two inch M.C.F., 65 litres/sec. fractionating oil diffusion pump backed by a "DAIA" 150 litres/sec. mechanical pump driven by a 3/4 horsepower, 220 V, 3 phase electric motor. The large electric motor eliminated any change of stalling the mechanical pump when a quench was made.

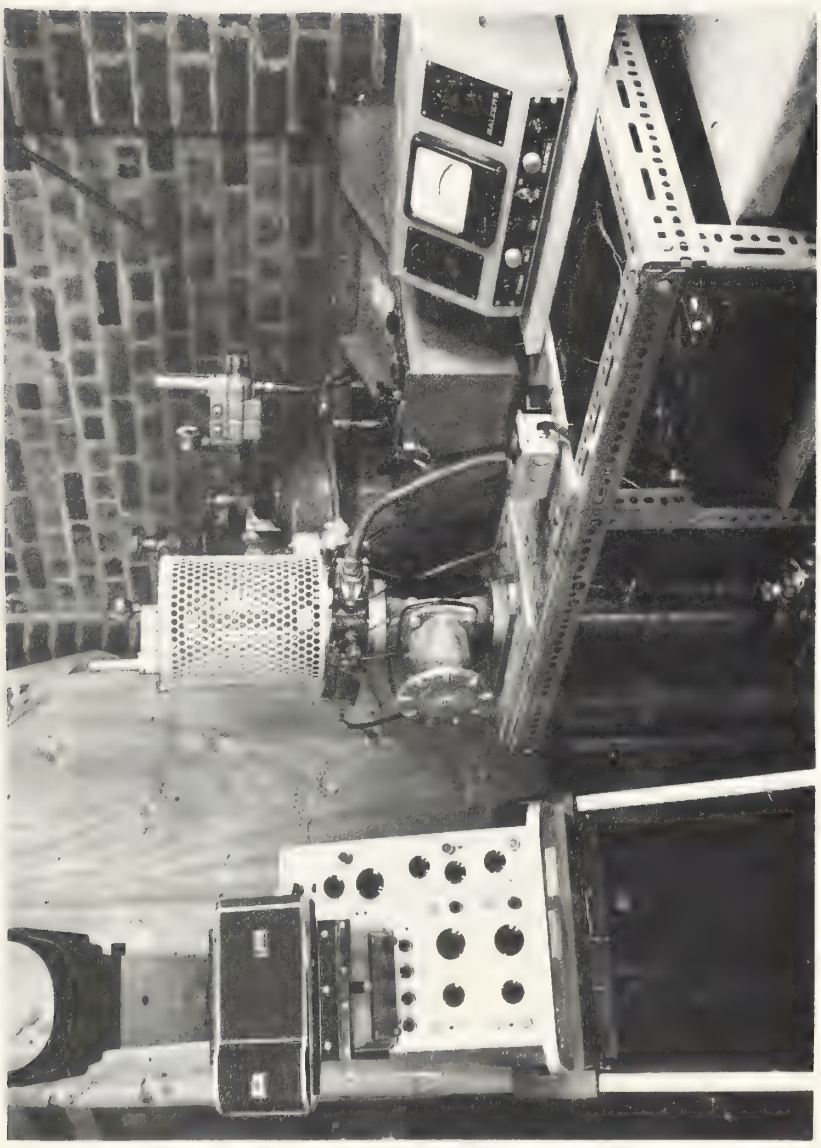

FIG. 6. EXPERIMENTAL APPARATUS COMPRISING A GAS QUENCH UNIT, VACUUM SYSTEM, AND DATA – RECORDING EQUIPMENT.

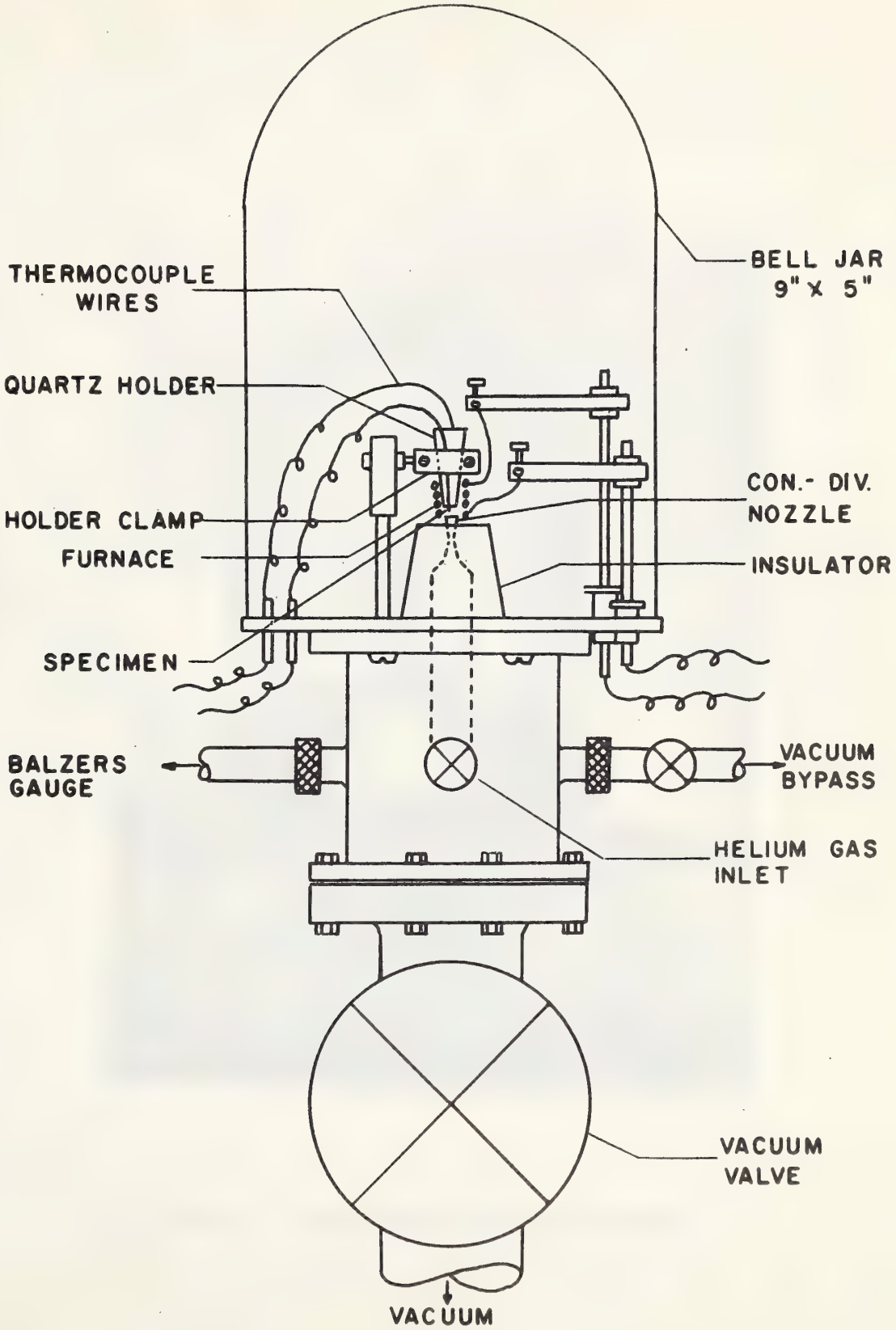

FIG. 7. SCHEMATIC DRAWING OF GAS QUENCHING APPARATUS
[NOT TO SCALE]

FIG. 8. GAS QUENCHING APPARATUS.

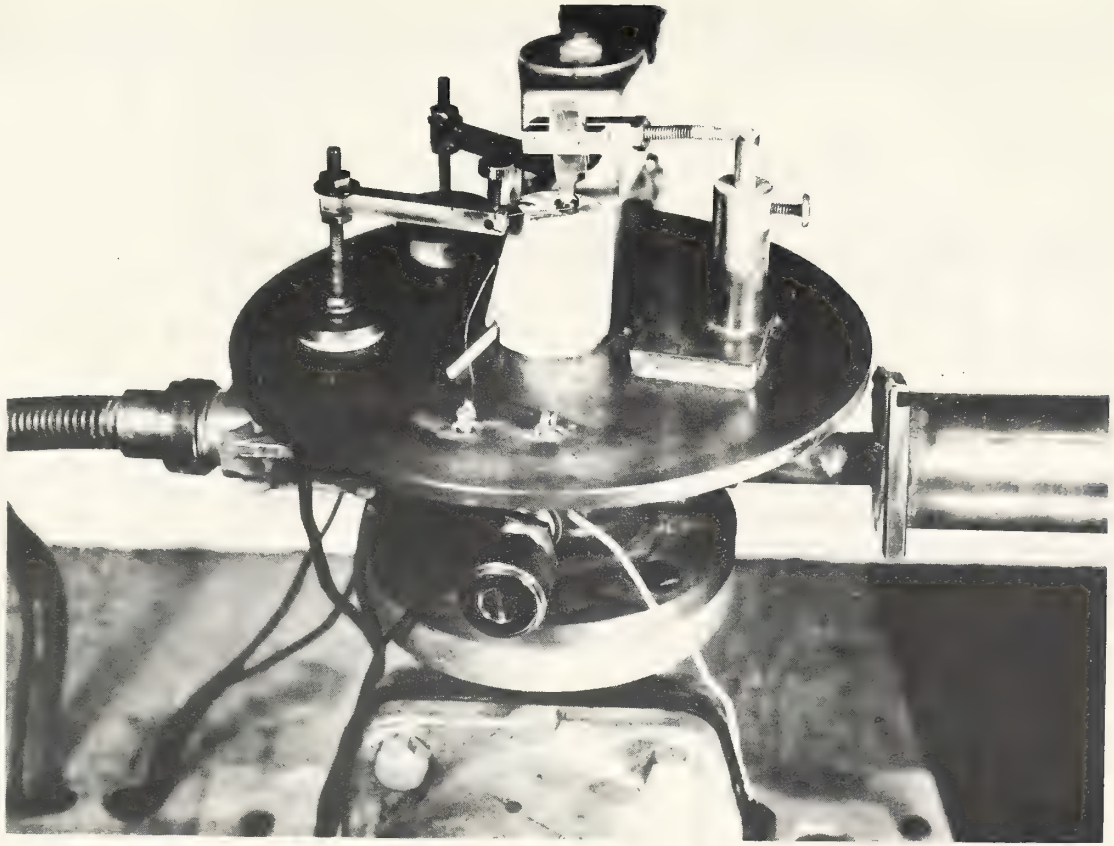

FIG. 9. GAS QUENCHING APPARATUS SHOWING THE SPECIMEN MOUNTED ON ITS QUARTZ HOLDER, SURROUNDED BY THE TUNGSTEN WIRE HEATER. [THE CERAMIC CRUCIBLE INSULATES THE NOZZLE FROM FURNACE HEAT].

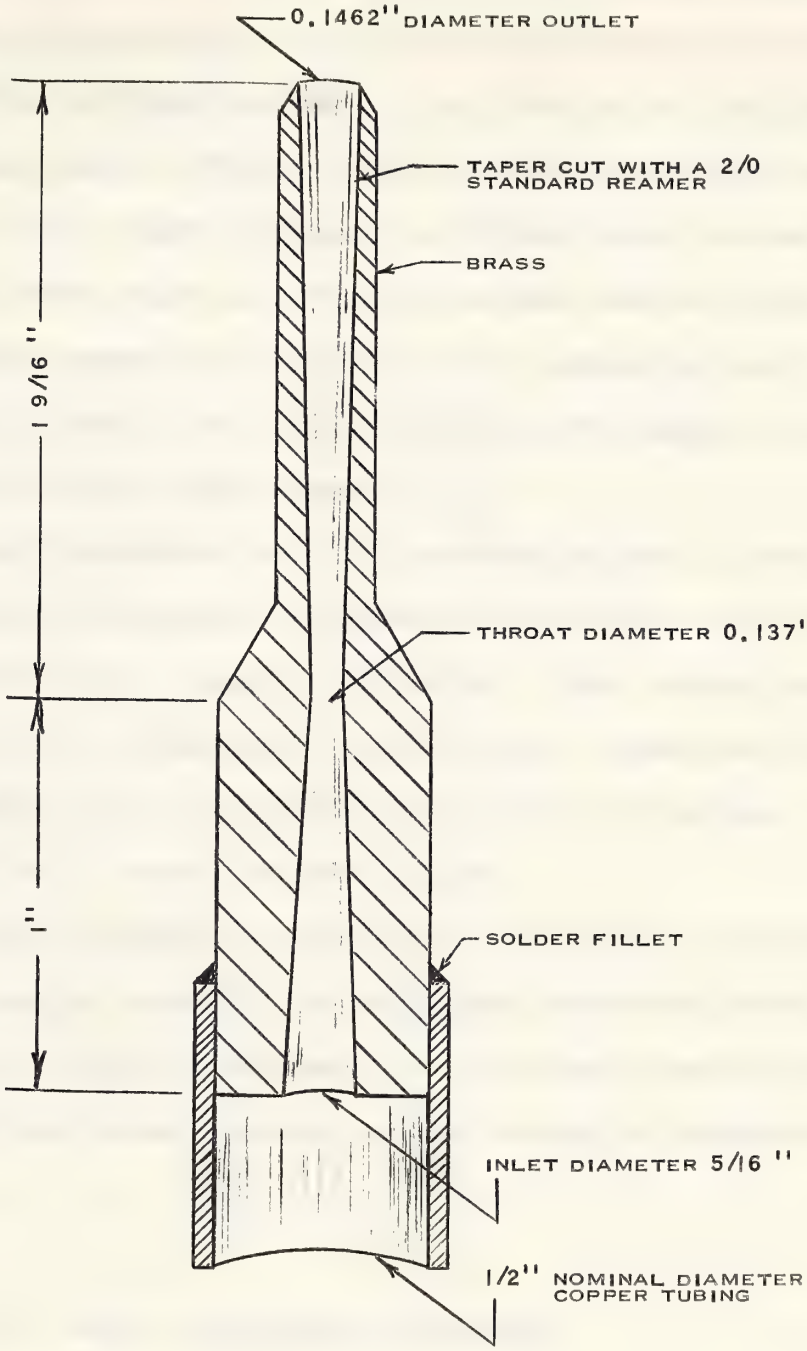

FIG. 10. CROSS – SECTION OF A CONVERGENT – DIVERGENT NOZZLE.
DESIGNED TO GIVE SUPERSONIC DISCHARGE VELOCITIES. NOT TO SCALE.

A Philips cold-cathode ionization gauge, type P.H.G. 90, connected to the base of the quenching enclosure, measured the system pressure. Calibration of the instrument was checked with a "Balzer" hot-cathode ionization gauge.

A 1 1/2 inch diameter cold trap* was mounted on the diffusion pump, with an "O" ring vacuum sealed joint to prevent any vapour from entering the quenching unit. On top of the cold trap a two inch "Speedivalve" separated the quenching and the vacuum units.

The quenching unit was mounted on the "Speedivalve". The auxiliary equipment was mounted on a two inch diameter by two inch long, flanged cylinder. Four vacuum sealed outlets on the cylindrical section were connected to the vacuum gauge, to a quenching valve, to a "roughing - out" line, and to a vacuum release valve.

The "roughing - out" line comprised a 1/2 inch flexible copper tube fitted with two "Speedivalves" and a copper tee. This arrangement facilitated bypassing the diffusion pump when evacuating the system of atmospheric gases.

(3) data - recording equipment

The thermocouple wires (in vacuo) were connected to the external leads by means of two "Kovar" glass - metal

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*It was not found necessary to use the cold trap to obtain a vacuum of 10^{-5} mm. Hg.

seals. These leads were then connected to the potentiometer and oscilloscope* via a two-pole double-throw toggle switch.

The potentiometer used was a "Leeds and Northrup" instrument reading to 0.025 m.v. The cold junction was at room temperature**, with maximum variation of $\pm$ 1 °C during an individual experiment.

The electrical current, produced by heating and cooling the thermocouple junction during an experiment, was fed into a "Tektronix" type 545A oscilloscope. The resulting cooling curve was then recorded on photographic film by a Polaroid Land camera fitted to the oscilloscope. Fig. 11 is an actual photograph taken during an experimental run.

C. Experimental Procedure

Specimens were prepared from the ingots of iron - 1, 2,3,5,7, and 10 at. pct. nickel which had been previously analysed*** for nickel content.

The specimen was mounted on the quartz holder and positioned within the furnace coil, which had been previously outgassed****, directly above the quench - nozzle orifice.

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*Better results were obtained by connecting the shielded oscilloscope leads directly onto the two "Kovar" glass - metal seals. This method seemed to cut down on interference.

**Close checks were kept on room temperature and the results of each experiment were corrected accordingly.

***These analyses confirmed previous data that the ingots were homogeneous and were in agreement with the calculated alloy percentages. See Appendix II for analyses.

****Newly - wound furnace coils were outgassed in a vacuum of 10^{-5} mm. Hg., by heating to a temperature of 1,000 °C and holding for a period of 5 minutes.

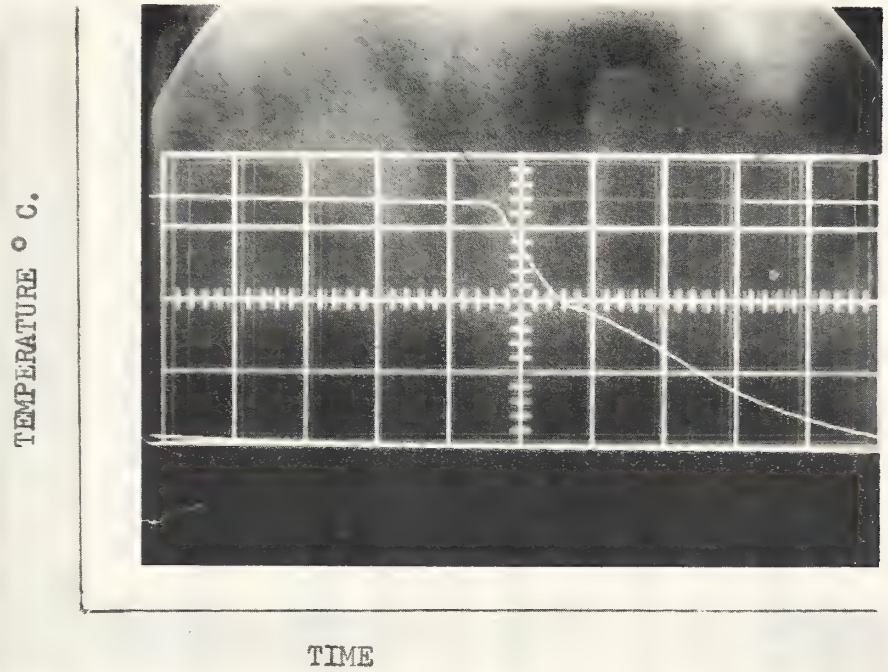

FIG. II. REPRESENTATIVE COOLING CURVE, RECORDED BY A POLAROID LAND CAMERA ATTACHED TO THE OSCILLOSCOPE.

The nozzle - to - specimen distance varied from zero to about one-quarter of an inch, depending upon the cooling rate desired (fastest rates were obtained by positioning the specimen within the nozzle orifice).

Evacuation of the system was started by simultaneously flushing the quench - gas lines to the system, and pumping through the "roughing - out" line. When a vacuum of 100 microns (minimum) had been attained, the system was opened to the diffusion pump circuit. Experiments were not conducted until the system had been evacuated to less than 10^{-5} mm. Hg.

Prior to an experiment, the oscilloscope was switched on and allowed a stabilization period of at least one hour. Further preparation consisted of selecting the proper time (horizontal) and temperature (vertical) scales on the oscilloscope, exposing the calibration grid*, checking the camera setting**, and choosing a convenient temperature datum***.

Room temperature was then recorded and the experiment begun by energizing the furnace coil until the specimen reached a temperature of 1,000 °C. The specimen was soaked at this temperature for one minute, and quenched by successively cutting the power to the furnace and closing the

---

*Exposure time of 5 seconds at a relative intensity setting of "11".
**Camera set on time exposure "B", at an aperture opening of f5.6 for type 47/speed 3,000 film.
***Datum selected by correlating the position of the cathode beam and the potentiometer reading until the desired temperature and beam position were attained.

solenoid valve switch. Quench gas was allowed to impinge upon the sample for a period of 2 or 3 seconds before closing the solenoid valve. During this interval of time, the cooling data were recorded on the Polaroid Land camera film by holding the shutter open on time exposure.

After each experiment, the pressure was reduced to less than 10^{-5} mm. Hg. before reheating prior to another quench on the same sample.

Polished specimens were transformed at various cooling rates and the surfaces examined microscopically for evidence of surface "rumple".

Figures 12 and 13 are photographs of the iron and brass clamp used for polishing samples on a Fisher "Vibro-matic" polisher.

Approximately 550 experiments were completed, using a total of about 60 different specimens.

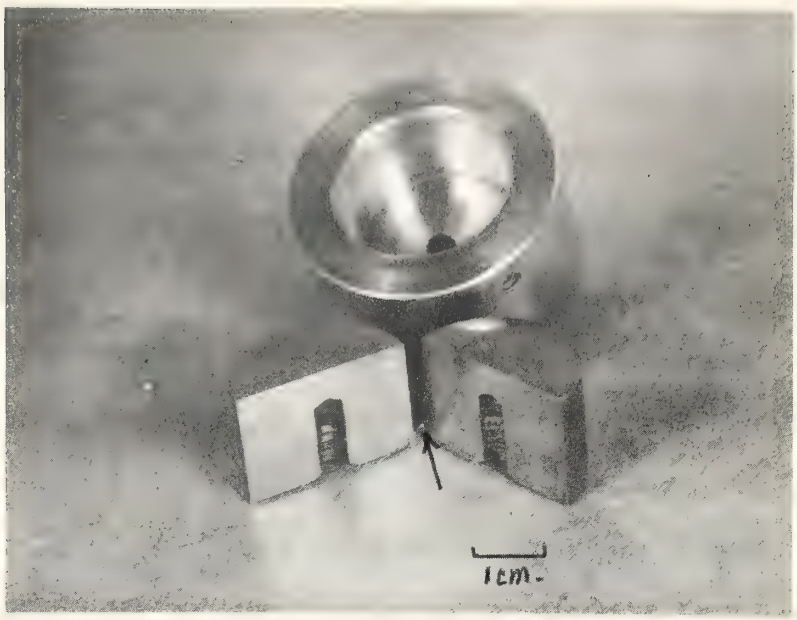

FIG. 12. DISMANTLED VIEW OF THE SPECIMEN POLISHING CLAMP. THE SPECIMEN IS SHOWN MOUNTED ON ITS THERMOCOUPLE WIRES.

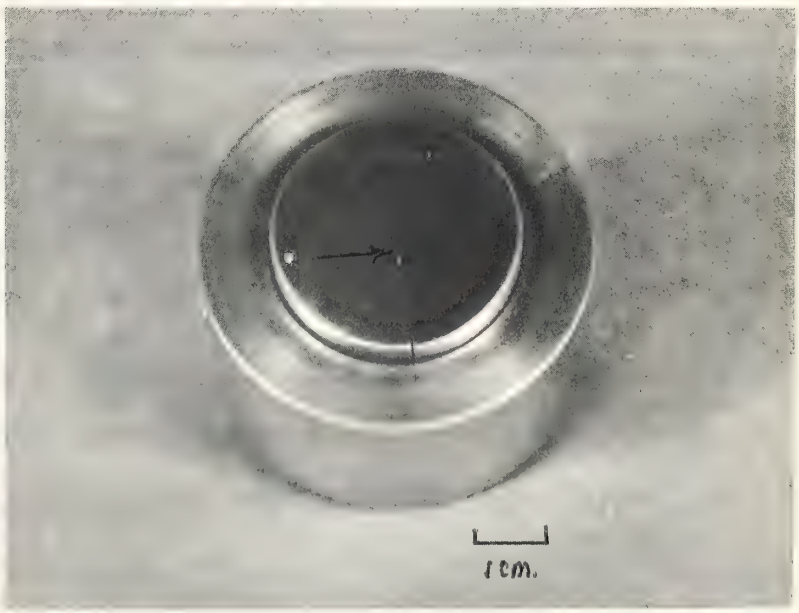

FIG. 13. ASSEMBLED VIEW OF THE SPECIMEN POLISHING CLAMP. THE SPECIMEN IS SHOWN MOUNTED BETWEEN THE IRON JAWS, READY FOR POLISHING.

RESULTS

The results on the measuremnt of transformation temperature vs. cooling rate are shown in Figures 14 to 17 inclusive. In the 1,2,3,5, and 7 at. pct. alloys, the transformation temperatures are depressed from an upper plateau to a lower plateau at critical cooling rates which vary with nickel content. The 10 at. pct. alloy appears to be devoid of this change in transformation temperature, and only one plateau exists. All the polished and transformed samples of the 10 at. pct. alloy displayed a surface "rumple", characteristic of martensite.

The curve for pure iron[29] is also shown in Fig. 14.

Because of the experimental difficulty involved in determining accurate values of critical rates, it is necessary to show these as "hatched" zones covering a cooling rate range of some 2,000 °C/sec.

Table VIII* is a summary of data taken from the curves in Fig. 14. Values in this table were used to plot the relationships shown in Figures 18 and 19.

Fig. 18 is a plot of transformation temperature vs. nickel content. Two nearlly-parallel curves are shown, representing the massive and martensitic transformation temperatures.

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*See Appendix IV.

Fig. 19 is a plot of nickel content vs. critical cooling rate. Each point on the curve is shown with the limits of error of plus or minus 1,000 °C/sec.

Figures 20 to 25 inclusive, show the microstructures of a few samples that were polished prior to quenching.

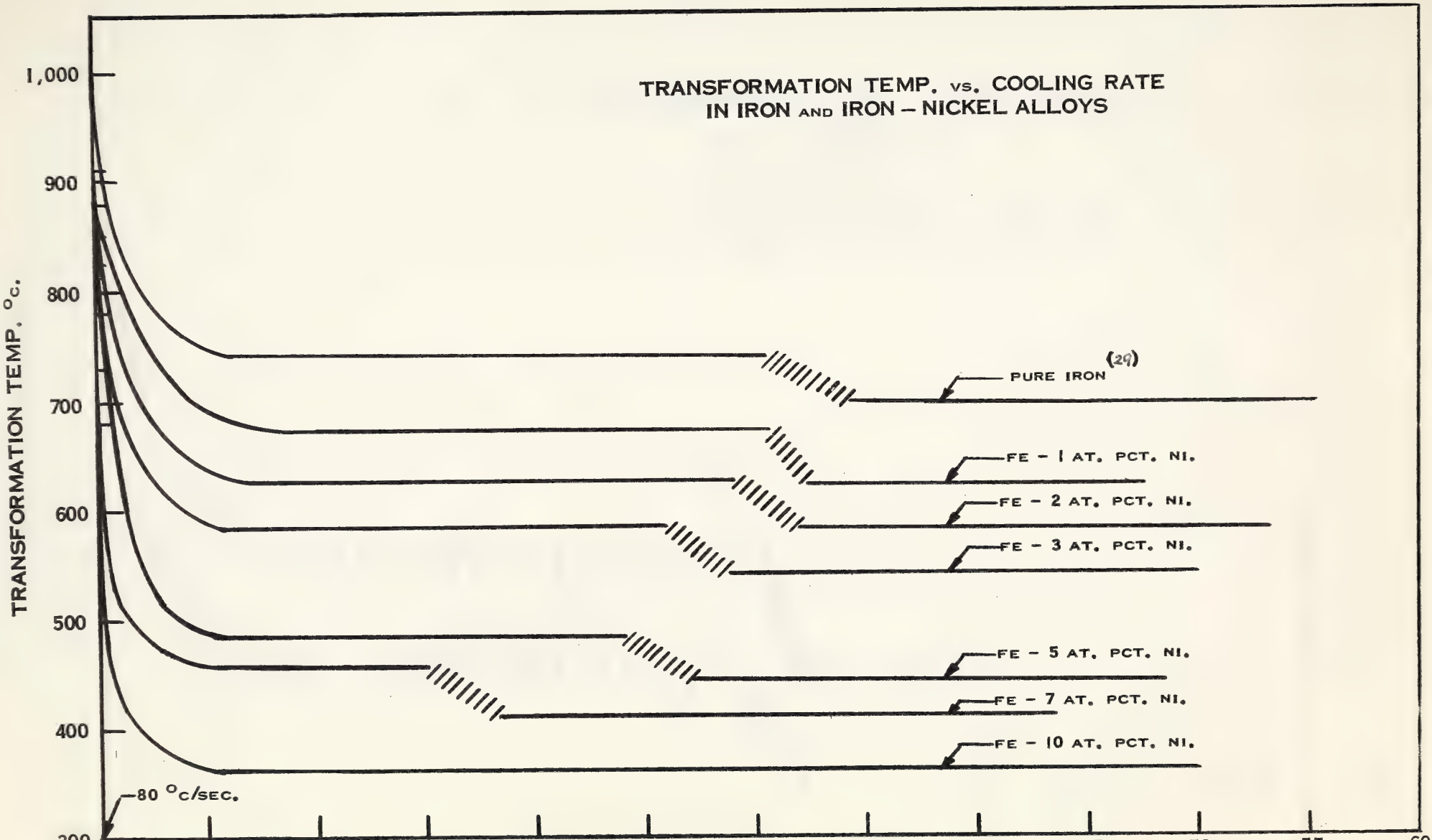
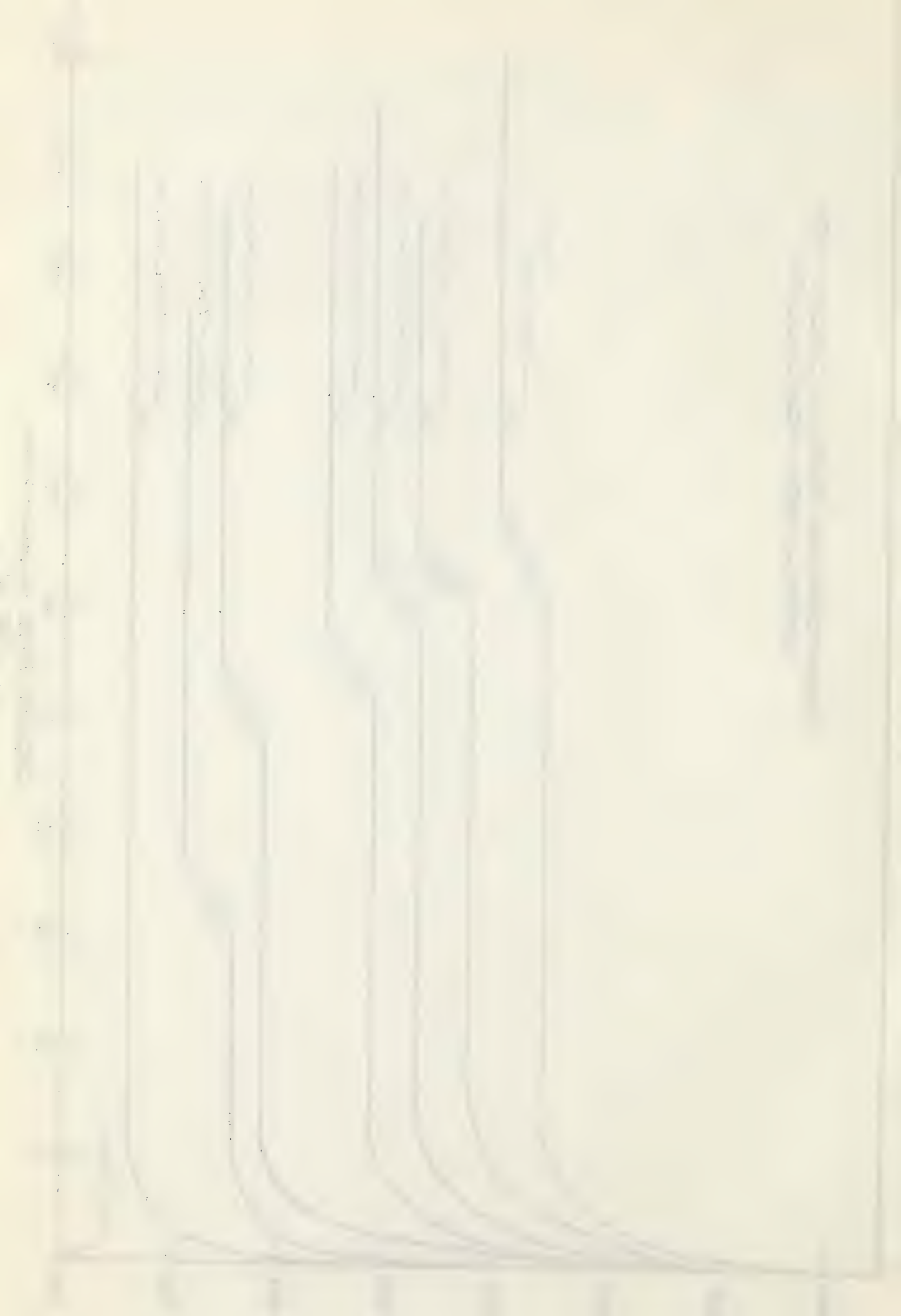

TRANSFORMATION TEMP. vs. COOLING RATE
IN IRON AND IRON – NICKEL ALLOYS
TRANSFORMATION TEMP. °C.
1,000
900
800
700
600
500
400
300
PURE IRON (24)
FE – 1 AT. PCT. NI.
FE – 2 AT. PCT. NI.
FE – 3 AT. PCT. NI.
FE – 5 AT. PCT. NI.
FE – 7 AT. PCT. NI.
FE – 10 AT. PCT. NI.
80 °C/SEC.
5
10
15
20
25
30
35
40
45
50
55
60
COOLING RATE THOUSANDS °C/SEC.

FIG. 14.



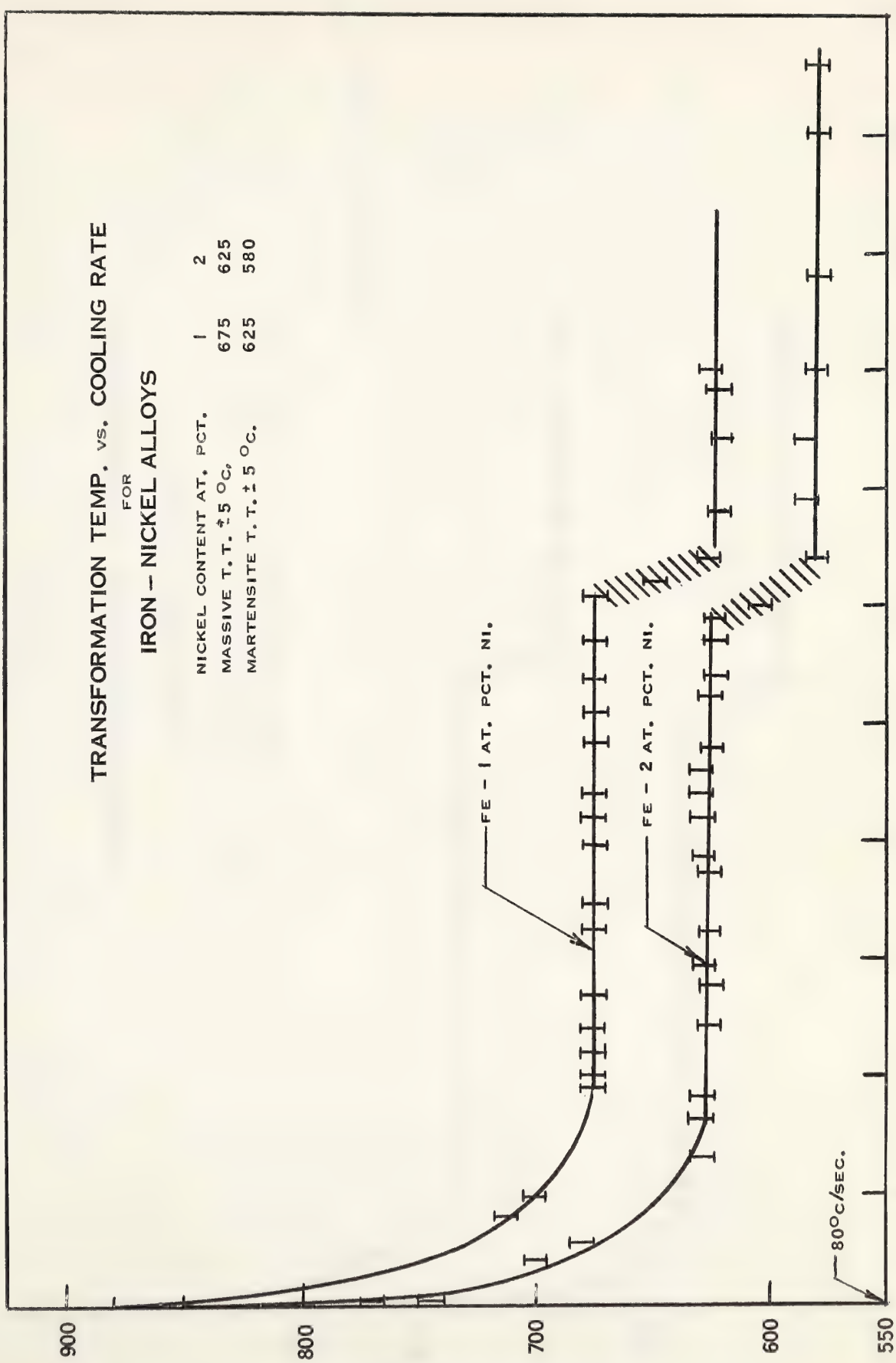

TRANSFORMATION TEMP. VS. COOLING RATE
FOR
IRON – NICKEL ALLOYS
NICKEL CONTENT AT. PCT. 1 2
MASSIVE T.T. ± 5 °C. 675 625
MARTENSITE T.T. ± 5 °C. 625 580
FE - 1 AT. PCT. NI.
FE - 2 AT. PCT. NI.
80°C/SEC.
TRANSFORMATION TEMP. °C.
900
800
700
600
550
5
10
15
20
25
30
35
40
45
50
55
COOLING RATE THOUSANDS °C/SEC.

FIG. 15.

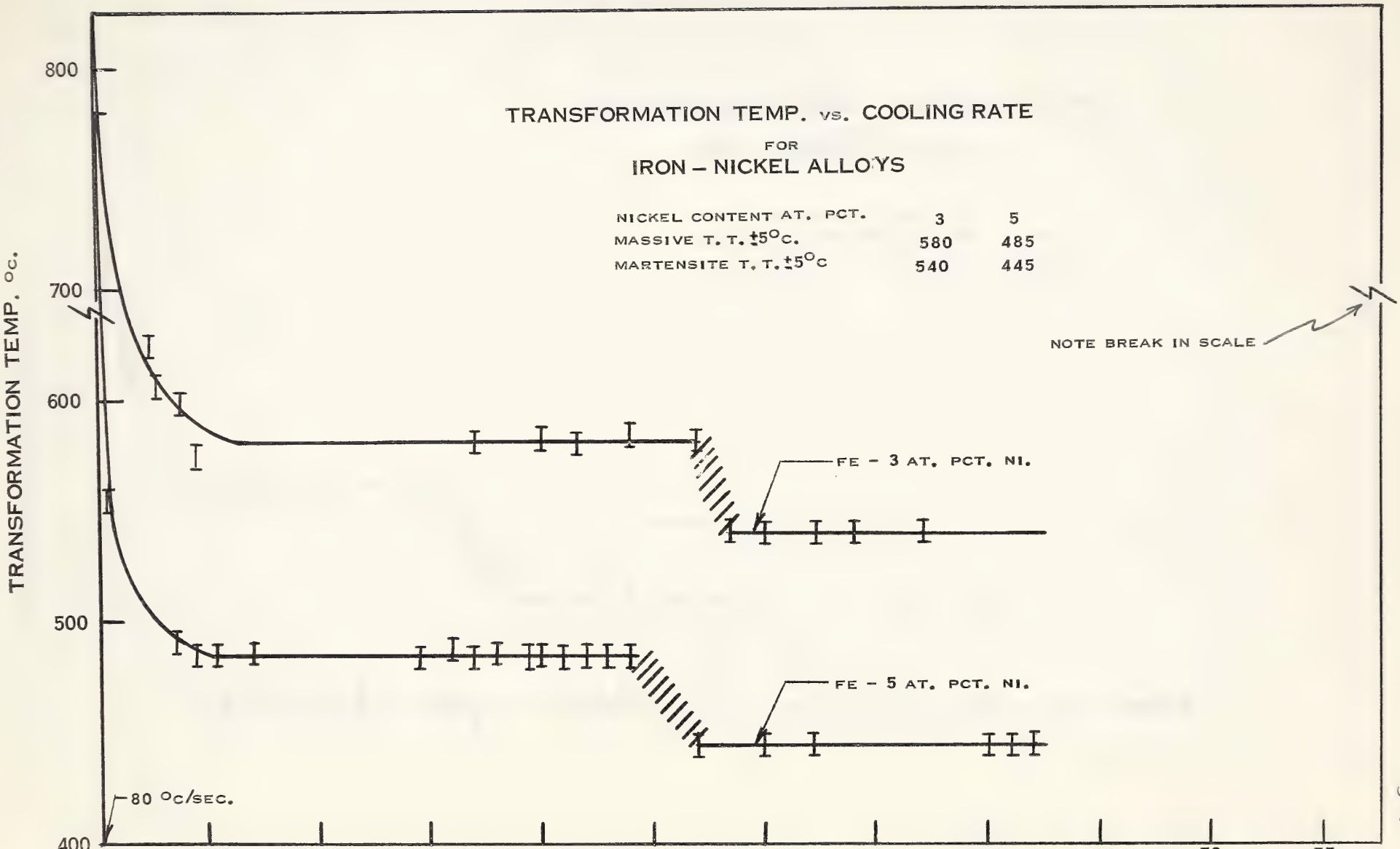

TRANSFORMATION TEMP. vs. COOLING RATE
FOR
IRON – NICKEL ALLOYS
NICKEL CONTENT AT. PCT. 3 5
MASSIVE T. T. ±5°C. 580 485
MARTENSITE T. T. ±5°C 540 445
NOTE BREAK IN SCALE
FE – 3 AT. PCT. NI.
FE – 5 AT. PCT. NI.
80 °C/SEC.
TRANSFORMATION TEMP. °C.
800
700
600
500
400
5
10
15
20
25
30
35
40
45
50
55
COOLING RATE THOUSANDS °C/SEC.

FIG. 16.

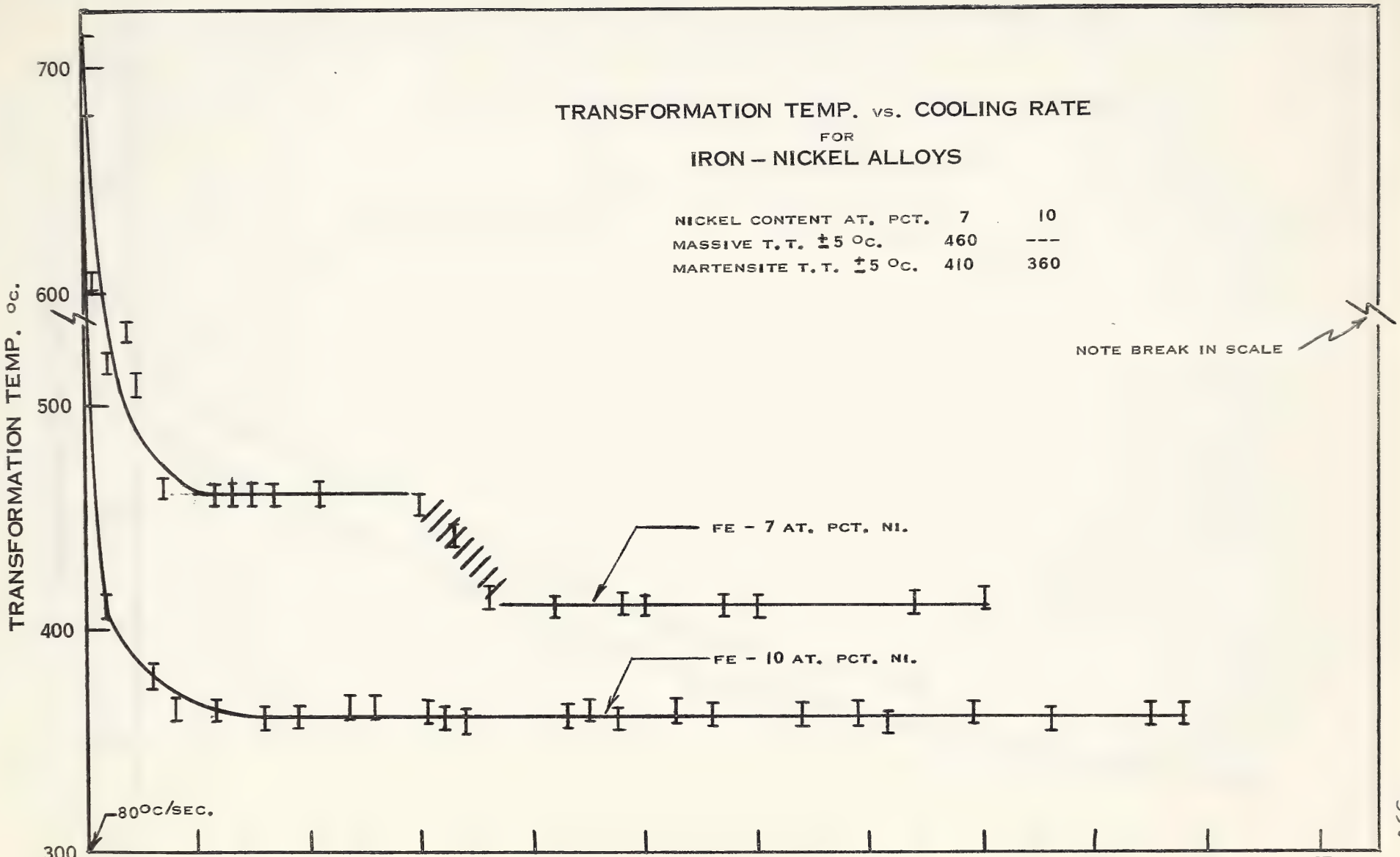

TRANSFORMATION TEMP. vs. COOLING RATE
FOR
IRON – NICKEL ALLOYS
NICKEL CONTENT AT. PCT. 7 10
MASSIVE T.T. ±5 °C. 460 ---
MARTENSITE T.T. ±5 °C. 410 360
NOTE BREAK IN SCALE
FE – 7 AT. PCT. NI.
FE – 10 AT. PCT. NI.
80°C/SEC.
TRANSFORMATION TEMP. °C.
COOLING RATE THOUSANDS °C/SEC.
700
600
500
400
300
5
10
15
20
25
30
35
40
45
50
55

FIG. 17.

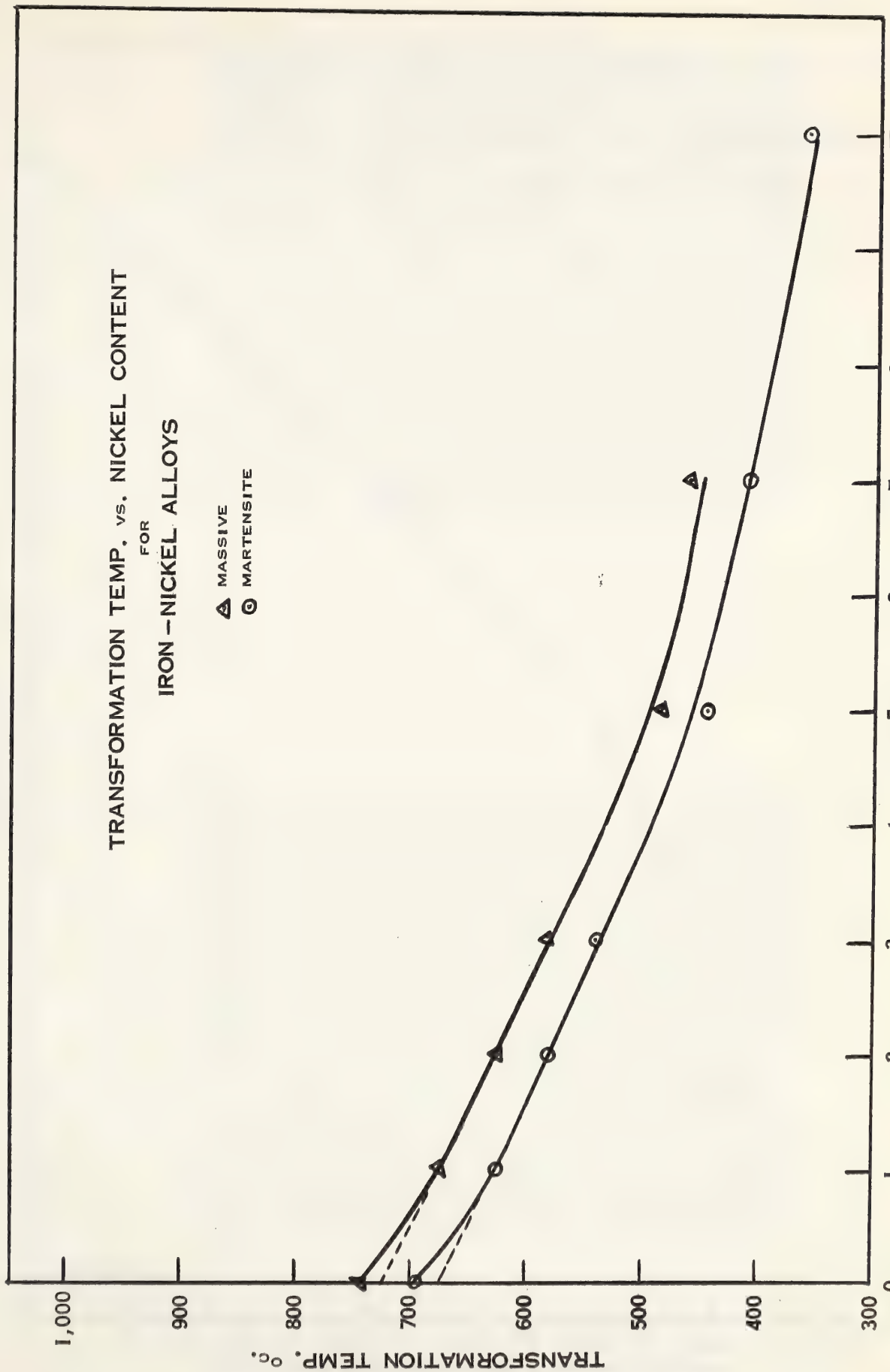

TRANSFORMATION TEMP. VS. NICKEL CONTENT
FOR
IRON – NICKEL ALLOYS
MASSIVE
MARTENSITE
TRANSFORMATION TEMP. °C.
1,000
900
800
700
600
500
400
300
0
1
2
3
4
5
6
7
8
9
10
NICKEL CONTENT AT. PCT.

FIG. 18.

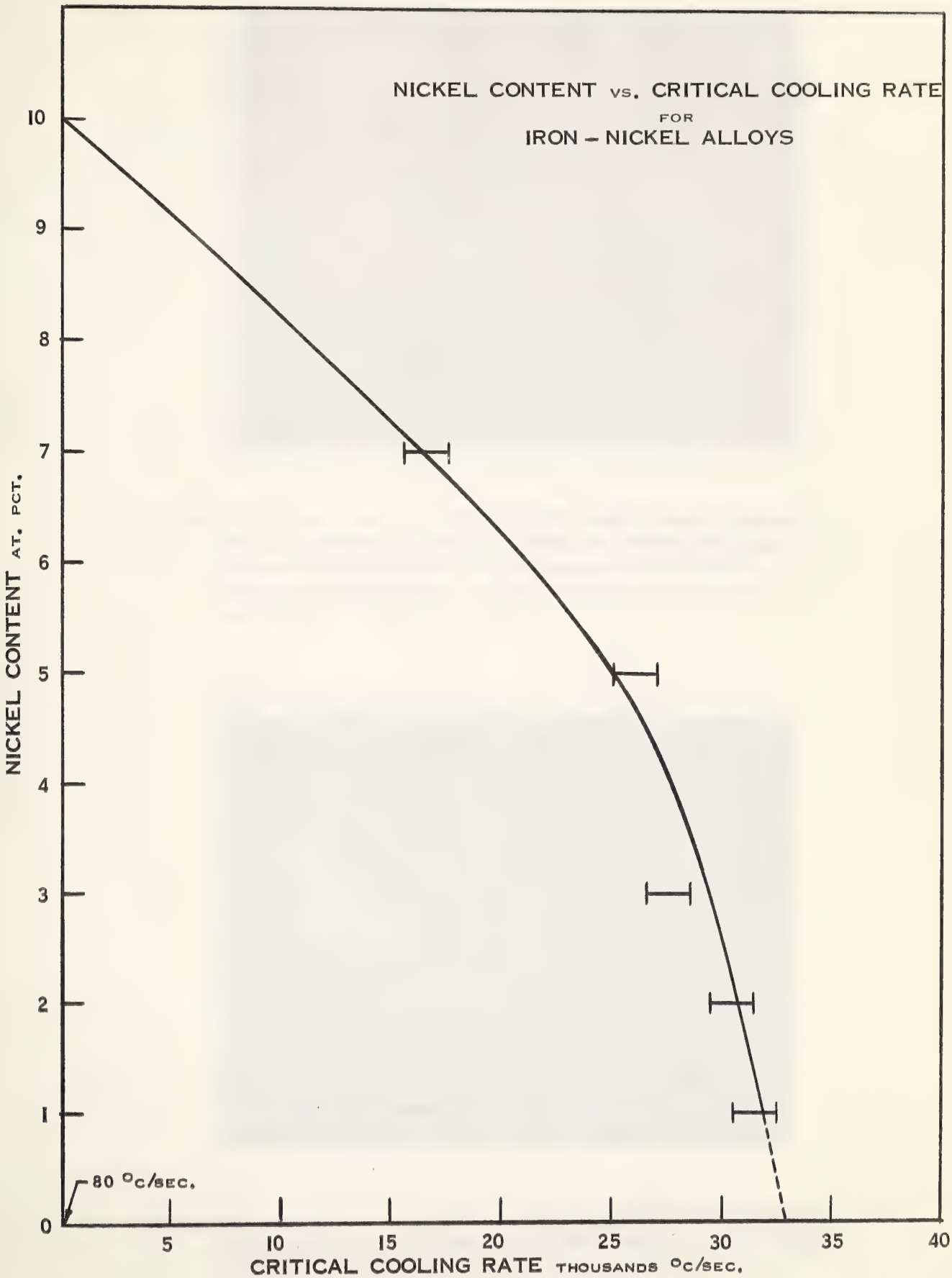

NICKEL CONTENT vs. CRITICAL COOLING RATE
FOR
IRON – NICKEL ALLOYS
NICKEL CONTENT AT. PCT.
10
9
8
7
6
5
4
3
2
1
0
80 °C/SEC.
5
10
15
20
25
30
35
40
CRITICAL COOLING RATE THOUSANDS °C/SEC.

FIG. 19.

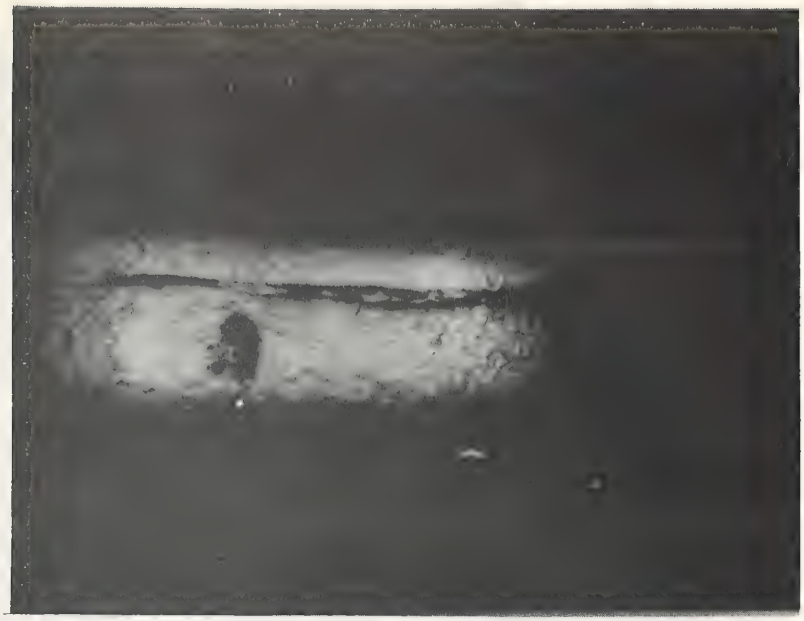


FIG. 20. FE – 2 AT. PCT. NI., POLISHED AND TRANSFORMED, X300. SURFACE HAS A MASSIVE APPEARANCE, [ALSO DISPLAYS A THERMAL ETCH]. QUENCHED FROM 1,000 °C AT A RATE OF 12,000 °C/SEC. TRANSFORMATION TEMPERATURE OF 626 °C.

FIG. 21. FE – 2 AT. PCT. NI., POLISHED AND TRANSFORMED, X300. SURFACE SHOWS A MARTENSITIC STRUCTURE. QUENCHED FROM 1,000 °C., AT A RATE OF 40,000 °C/SEC. TRANSFORMATION TEMPERATURE OF 580 °C.

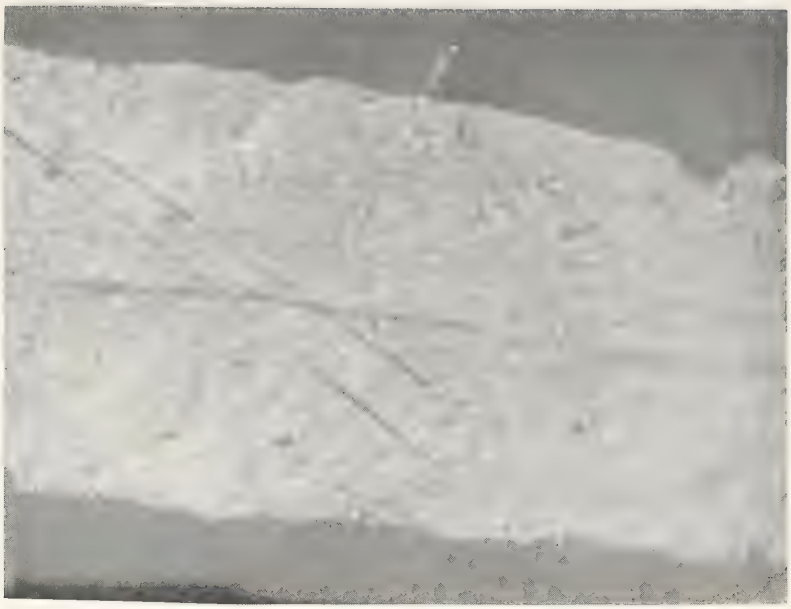


FIG. 22. FE – 7 AT. PCT. NI., POLISHED AND TRANSFORMED, X300. SURFACE HAS A MASSIVE APPEARANCE.
QUENCHED FROM 1,000 °C., AT A RATE OF 7,500 °C/SEC.
TRANSFORMATION TEMPERATURE OF 460 °C.

FIG. 23. FE – 7 AT. PCT. NI., POLISHED AND TRANSFORMED, X300. SURFACE SHOWS A MARTENSITIC STRUCTURE.
QUENCHED FROM 1,000 °C., AT A RATE OF 30,000 °C/SEC.
TRANSFORMATION TEMPERATURE OF 410 °C.

FIG. 24. FE – 10 AT. PCT. NI., POLISHED AND TRANSFORMED, X300. SURFACE SHOWS A MARTENSITIC STRUCTURE. QUENCHED FROM 1,000 °C., AT A RATE OF 80 °C/SEC. TRANSFORMATION TEMPERATURE OF 430 °C.

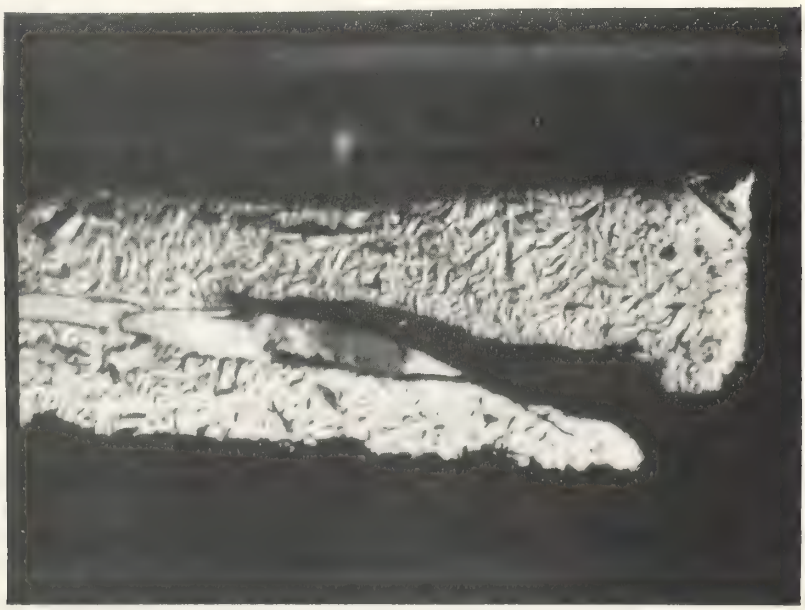


FIG. 25. FE – 10 AT. PCT. NI., POLISHED AND TRANSFORMED, X300. SURFACE SHOWS A MARTENSITIC STRUCTURE. QUENCHED FROM 1,000 °C., AT A RATE OF 48,000 °C/SEC. TRANSFORMATION TEMPERATURE OF 362 °C.

DISCUSSION

The effect of cooling rate on the transformation temperature in iron[29], and iron - nickel alloys is shown in Figures 14, 15, 16, and 17. In each of the alloys examined, excepting the 10 at. pct. alloy, there is a critical cooling rate at which depression of the transformation temperature occurs. The value of the critical rate decreases with increased nickel content. This implies that for each alloy there exists a critical cooling rate, above which the quenched sample is martensitic, and below which the quenched material is massive, the criterion being that surface rumpling is evident on the martensitic sample and absent on the massive sample.

The 10 at. pct. nickel samples exhibit well-defined surface rumpling over the complete range of cooling rates utilized, suggesting absence of the massive transformation. This contradicts results obtained by Owen and Gilbert[3], who in their discussion, state that iron - nickel alloys, of less than 15 pct. nickel, transform to massive alpha on cooling at rates as fast as 5,000 °C/sec. Figures 24 and 25 are photographs showing the microstructures of two iron - 10 at. pct. nickel alloy samples quenched at rates of 80 and 48,500 °C/sec. respectively. These offer evidence of a martensitic transformation at cooling rates far below that of 5,000 °C/sec.

Owen, in a discussion[30] of a paper written by Owen and Gilbert[31], states that martensite plates cannot be distinguished in quenched alloys of less than 10 at. pct. nickel. Figures 21 and 23 bear evidence to the fact that martensite can be formed in alloys of less than 10 at. pct. nickel, provided the samples are subjected to sufficiently rapid cooling rates.

Fig. 18 is a plot relating transformation temperature to nickel content, and shows a nearly-linear relationship between 1 and 5 at. pct. nickel for both the massive and martensitic transformations. Below 1 at. pct. nickel the curves bend slightly upward to values of 695±10 °C and 740±10 °C (from M.J. Bibby[29]) respectively. Extrapolation of the linear portion of the M_s vs. Ni content curve yields a transformation temperature about 680±5 °C for zero pct. nickel*, which is in relatively close agreement with M.J. Bibby's Value of 695±10 °C.

The slopes of the curves in Fig. 18 change about 5 at. pct. nickel, with the martensitic transformation curve continuing on a linear path of lower gradient, to the 360±5 °C M_s value of the 10 at. pct. alloy. It would seem presumptuous at this time to show the massive transformation curve as blending with the martensitic transformation curve to become one near the 10 at. pct. nickel content. Therefore,

---

*That is, at zero pct. nickel, iron containing 0.009 wt. pct. carbon has an M_s value about 680±5 °C.

this curve has been shown to stop at the last value recorded in the experiments.

Subsequent to the values determined for low carbon iron (see Fig. 14), M.J. Bibby[29] conducted a number of experiments on high purity iron containing less than 0.0015 pct. carbon and obtained an M_s temperature value of 750 ± 10°C. These results prompted a study into the effect of higher carbon content and led to the conclusion that the M_s temperature in iron varies from 750 ±10°C to 540 ±10°C for carbon content ranging from 0.0015 to a postulated 0.03 pct. respectively. This implies that the value of M_s varies inversely with carbon content of the samples. If this postulate is applied to the iron - nickel system, it does not seem presumptuous to suggest that if the samples contain much less than 0.009 pct. carbon, the M_s temperature values will be somewhat higher than those reported in Fig. 14. Supposing this to be the case, one might find that the M_s values in Fig. 18 would extrapolate to a zero pct. nickel value in the order of 750 °C, instead of 680 °C.

In addition to a consideration of the results obtained when investigating the effect of carbon content on the transformation temperature, it seems worthwhile to keep in mind the soaking time and soaking temperature. A number of samples, each of a different nickel content, were quenched after being soaked for various times (10 sec. to 10 min.) and at various temperatures. The data imply that

neither soaking time nor soaking temperature (935 °C to 1,100 °C) has any effect on the value of M_s.

The value of M_s for pure iron has been a subject of much controversy for many years. If the experimental values from Cohen and Kaufman's[1] paper are extrapolated to zero pct. nickel, an M_s value of 1,000 °K (727 °C) is obtained. Owen and Gilbert[3] show a value of about 560 °C, based on M_s values extrapolated from Fe-Ni-C data by Stevens and Haynes formula[32]. Opposing both of these values is the long-accepted M_s value of 520 °C for pure iron[33].

From the previous discussion it would appear that any one of these values could possibly be correct -- depending upon the carbon content of the sample. However, only Owen and Gilbert[3] recognize the existence of two transformation products, one being of a massive* nature and the other martensitic. On the other hand, Cohen and Kaufman[1] assume that the complete range of iron - nickel alloys transform martensitically, with no mention of cooling rates, or of the existence of the massive transformations. One might therefore suggest that the thermodynamic theories presented by these researchers[1] often apply to the massive transformation instead of the martensitic transformation.

Fig. 26 is the plot of Fig. 18 with transformation temperature curves, obtained or calculated by previous

---

*The term "massive" was used as early as 1939 by Greninger[34] and again in 1955 and 1958 by Hull and Garwood[35], and Massalski[36], resp., to describe a type of transformation product.

researchers, superimposed upon it. The M_s temperature curve from Owen and Gilbert[3] is in reasonably close agreement with my experimental values, in the range of 10 to 5 at. pct. nickel, but departs quite markedly in the 4 to 0 at. pct. nickel zone. However, the massive transformation temperature curve largely disagrees with my values; departures range from 80 °C to about 120 °C. The curve from Cohen and Kaufman[1] (for experimental values of M_s) follows closely the massive temperature plot of Owen and Gilbert[3], but is in obvious disagreement with my experimental values of M_s temperature. Extrapolation of the Cohen and Kaufman plot, to zero pct. nickel, yields a value about 1,000 °K (727 °C), which is approximately the same as the value obtained by extrapolating my massive transformation temperature curve to zero pct. nickel. A fourth comparison is shown near the bottom of Fig. 26. This plot is based on an empirical formula*, offered in many textbooks, and shows reasonable agreement with my experimental values, in the 5 to 10 at. pct. nickel range, but diverges in the lower nickel region to a final M_s value of 500 °C at zero pct. nickel.

Fig. 19 relates nickel content to the critical cooling rate at which a depression from the massive transformation temperature to the M_s temperature takes place. This plot is approximately linear from 5 to 1 at.

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*$M_s = 500 - 17\ (\%\ Ni)\ °C$[37]

pct. nickel. Extrapolation to zero pct. nickel yields a critical rate of approximately 33,000±1,000 °C/sec. for pure iron. This value agrees with the experimental value recorded by M.J. Bibby[29]. Linearity of the relationship is lost in the 5 to 10 at. pct. nickel range due to the appearance of martensite in the 10 at. pct. alloy cooled at rates below 80 °C/sec.

The critical cooling rate for the production of martensite may be simply that rate required to prevent diffusion transformations. However, the subject may be more involved than this -- although this has yet to be shown. If the critical cooling rate is no more than that rate required to prevent a transformation by diffusion, then the reduction in critical rate with increased nickel content is a reasonable consequence. The rate of transformation (by diffusion) of austenite to ferrite is reduced considerably by nickel additions[38] and is of course, the basis upon which the design of "hardenable" nickel alloy steels depends. There are, however, insufficient data to permit a quantitative analysis of the relationship between critical cooling rate and nickel content.

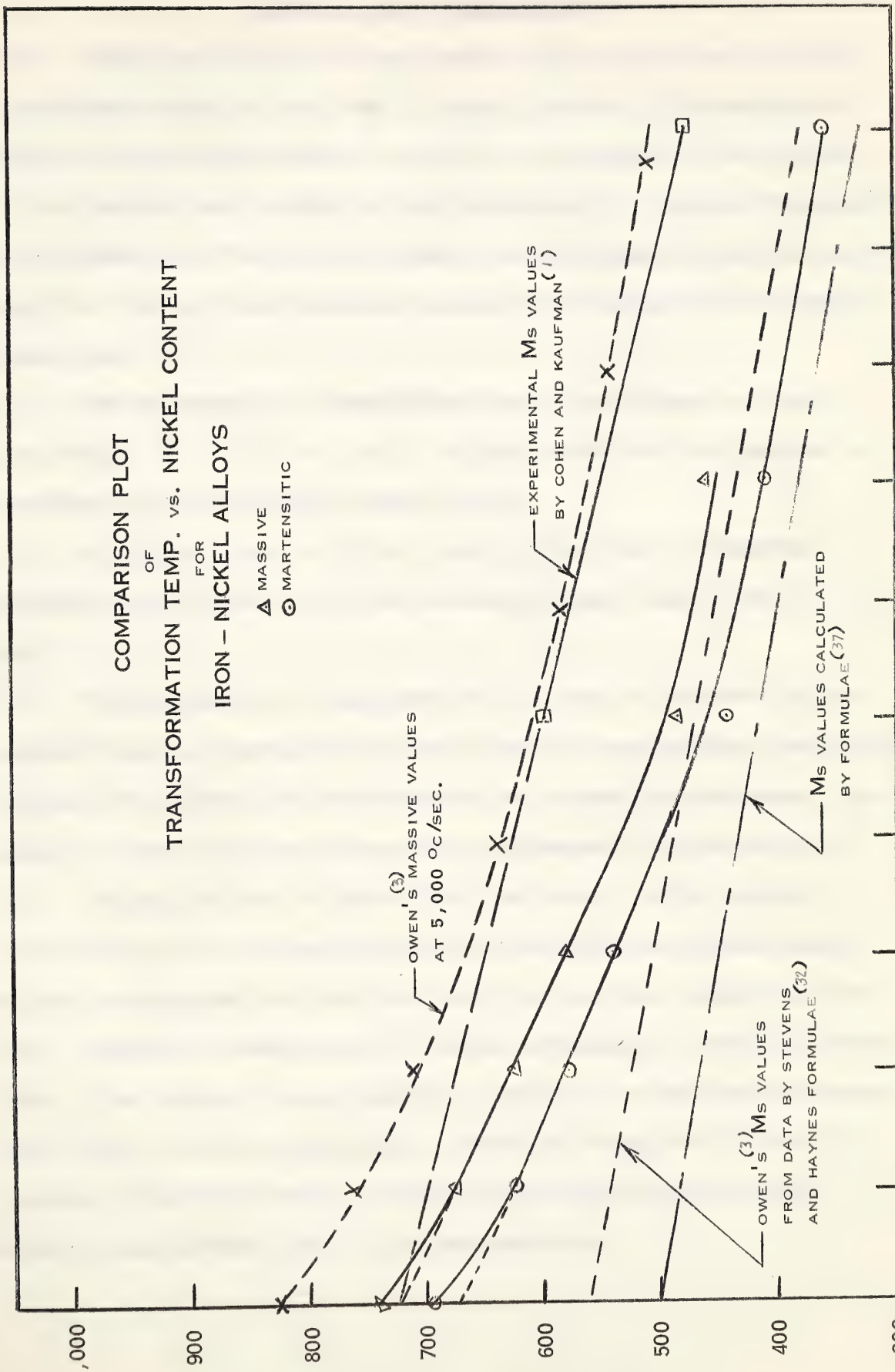
COMPARISON PLOT
OF
TRANSFORMATION TEMP. vs. NICKEL CONTENT
FOR
IRON – NICKEL ALLOYS
△ MASSIVE
⊙ MARTENSITIC
EXPERIMENTAL Ms VALUES BY COHEN AND KAUFMAN(1)
OWEN'S(3) MASSIVE VALUES AT 5,000 °C/SEC.
Ms VALUES CALCULATED BY FORMULAE(37)
OWEN'S(3) Ms VALUES FROM DATA BY STEVENS AND HAYNES FORMULAE(32)
TRANSFORMATION TEMP. °C.
1,000
900
800
700
600
500
400
300
0
1
2
3
4
5
6
7
8
9
10
NICKEL CONTENT AT. PCT.

FIG. 26.

SUMMARY AND CONCLUSIONS

(1) There are two different mechanisms operative in phase transformations in the iron - nickel system. The one operative at the lower rates of cooling is presumably a process of nucleation and growth by diffusion and is called "massive" transformation. The other, operative at higher rates of cooling, is a shear type process called the "martensitic" transformation.

(2) The martensitic transformation takes place in iron - low nickel (0 to 10 at. pct.) alloys provided the sample is quenched at sufficiently high rates.

(3) Alloys of iron - 10 at. pct. nickel will transform martensitically at very slow cooling rates (less than 80 °C/sec.).

(4) The value of M_S temperature in iron is lowered by increased nickel content. The fact that M_S approaches the equilibrium transformation temperature as nickel is increased implies a lower driving force with increased solute content.

(5) Extrapolation of data from the iron - high nickel (30 to 15 pct.) system does not agree with the experimental values determined in this project for the low nickel alloys.

(6) Previous researchers[1] have erroneously labelled their iron - low nickel transformation products as martensite when they were, in all probability, massive. Owen and Gilbert[3] however, classify their product of slow cooling rates as massive and assume that the martensitic

transformation will not occur in the low nickel alloys. Had Owen and Gilbert[3] subjected their samples to higher rates of cooling (at least 30,000 °C/sec.) they would have produced martensitic transformations.

A future associated project might be undertaken to investigate the effects of varying carbon content, in iron - nickel alloys, on the massive and martensitic transformation temperatures, and the critical cooling rates.

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APPENDIX I.

CIRCUIT DIAGRAM OF ELECTRIC ARC FURNACE.

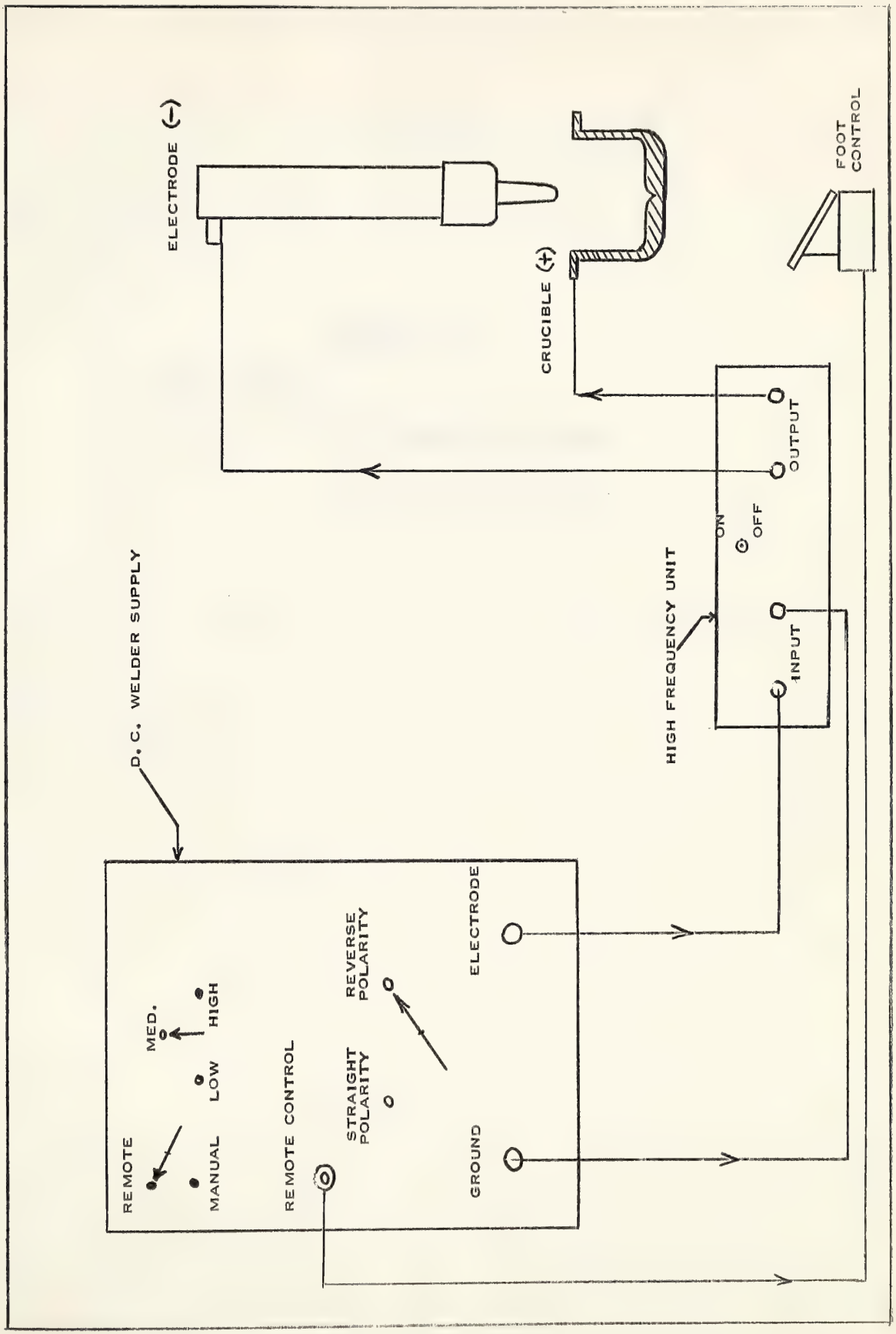

FIG. 27. CIRCUIT DIAGRAM FOR ELECTRIC ARC FURNACE.

APPENDIX II

ANALYSES OF:

(a) materials used.

(b) alloys prepared.

(a) materials used

IRON SOURCE

From: CRUCIBLE STEEL COMPANY OF AMERICA,
SYRACUSE, N.Y., U.S.A.

Customs order number 04384C
Mill order number 4-29-200 10-9
Invoice number 2648
Shipped from Malcomb Toronto Branch 3/20/59.

FERROVAC E CD NATURAL 5/8 inch Rod Heat No. 04264

CHEMICAL ANALYSIS: (pct.)

.009 C	.001 Mn	.002 P	.007 S	.006 Si
.04 Ni	.01 Cr	.004 V	.01 Mo	.007 Co
.014 Cu	.01 Al	.0003 N	.008 O	.00006 H
.0004 Pb				

NICKEL SOURCE

SPECTROGRAPHICALLY STANDARDIZED MATTHEY NICKEL RODS

5 mm. Diameter X 15 cm. Long

Laboratory No. 23226

Catalogue No. J.M. 890

JOHNSON, MATTHEY & CO. LTD.
73/83 HATTON GARDEN,
LONDON E.C.I.

ANALYSIS: pct.

.001 Fe, .001 Si, .0003 Al, .0002 Ca, .0002 Mg, .0001 Cu, less than .0001 of Mn or Ag.

The following elements were sought but not detected: As, Au, B, Ba, Be, Bi, Cd, Co, Cr, Cs, Ga, Ge, Hf, Hg, In, Ir, K, Li, Mo, Na, Nb, Os, P, Pb, Pd, Pt, Rb, Re, Rh, Ru, Sb Se, Sn, Sr, Ta, Te, Ti, Tl, V, W, Zn, Zr.

(b) alloys prepared

Samples cut from test ingots of iron - 20 at. pct. nickel, (prepared in Electric Arc Furnace) to determine homogeneity of the specimens.

Wet Chemical Analysis (U of A laboratory, Department of Mining & Metallurgy).

Sample #1	19.15	at. pct. Ni.
Sample #2	19.32	"
Sample #3	19.35	"
Sample #4	19.70	"

Sample #2 sent out for SPECTROGRAPHIC ANALYSIS at:

CHICAGO SPECTRO SERVICE LAB. INC.
2454 West 38 St.,
CHICAGO 32, ILLINOIS.

ANALYSIS: Molybdenum Approx. 0.01 pct.
Chromium Approx. 0.01 pct.

"Spectrographic examination did not reveal the presence of any other elements greater than 0.01 pct. Copper, Silicon, Manganese were detected but were less than the 0.01 pct. level".

Samples of drillings (approx. 5 gms. each) from the 15, 10, 7, 5, and 2 atomic pct. ingots were sent to:

TECHNICAL SERVICE LABORATORIES,
355 KING ST. W.,
TORONTO 2B, ONTARIO.

ANALYSIS: Report No. C-21012-4 T-4982

15.02 atomic pct. Ni.
10.15 "
7.10 "
5.20 "
1.92 "

APPENDIX III.

TABULATION OF TRANSFORMATION TEMPERATURES ON COOLING.

TABLE I

Transformation Temperatures of pure iron*

Cooling rate °C/sec.	Transformation temp. ± 10 °C.
0	910
300	880
800	845
2,000	802
5,000	740
10,000	740
15,000	740
20,000	735
25,000	735
30,000	733
32,000	721
36,000	709
40,000	695
47,000	695
55,000	695

*From M.J. Bibby(29)

TABLE II

Transformation Temperatures of
Iron - 1 at. pct. Nickel.

Cooling rate °C/sec.	Transformation temp. ± 5 °C.	Cooling rate °C/sec.	Transformation temp. ± 5 °C.
0	880	19,800	674
150	817	21,000	676
175	782	21,700	674
190	770	22,000	676
4,000	713	24,200	674
4,400	706	25,500	675
4,400	702	26,900	675
4,800	702	27,400	674
9,700	676	28,500	675
10,000	676	29,400	675
11,000	676	30,300	674
11,300	674	31,000	650
11,500	675	32,000	627
11,800	666	34,000	622
12,000	676	34,600	617
12,700	674	37,100	621
13,400	676	39,400	623
16,200	676	40,000	624
17,000	666	40,000	626
17,300	674		

TABLE III

Transformation Temperatures of
Iron - 2 at. pct. Nickel.

Cooling rate °C/sec.	Transformation temp. ± 5 °C.	Cooling rate °C/sec.	Transformation temp. ± 5 °C.
0	850	19,300	628
100	767	21,000	630
150	744	22,000	630
1,700	700	23,000	630
1,900	700	24,000	625
2,100	700	26,200	626
2,800	682	27,000	624
3,200	680	28,500	624
6,400	628	29,000	627
6,900	628	29,500	624
8,100	628	30,000	604
8,200	628	32,000	580
8,600	628	32,300	594
8,800	628	32,300	582
9,100	628	34,600	585
12,100	626	37,000	587
13,800	625	40,000	582
14,700	628	44,000	580
16,200	626	50,000	580
18,600	626	53,000	580

TABLE IV

Transformation Temperature
of Iron - 3 at. pct. Nickel.

Cooling rate °C/sec.	Transformation temp. ± 5 °C.
0	825
145	678
2,400	624
2,700	612
3,900	598
4,000	600
4,400	575
4,800	600
17,000	580
20,000	588
21,600	580
22,000	577
22,000	585
24,000	585
24,200	586
27,000	582
28,500	542
30,000	540
32,300	543
34,000	541
34,600	543
37,200	543

TABLE V

Transformation Temperatures of
Iron - 5 at. pct. Nickel.

Cooling rate °C/sec.	Transformation temp. ± 5 °C.	Cooling rate °C/sec.	Transformation temp. ± 5 °C.
0	780	19,400	485
65	636	20,000	485
180	632	21,000	484
200	628	22,000	485
250	600	23,000	486
900	555	24,000	484
1,100	555	24,200	486
3,700	491	24,400	486
4,400	485	27,000	444
5,400	486	30,000	445
7,000	487	32,200	445
14,700	484	34,600	444
15,600	488	37,000	446
16,000	488	37,200	444
16,200	484	40,000	443
16,700	488	40,000	446
17,300	485	41,000	443
18,000	487	42,000	446
18,600	486		

TABLE VI

Transformation Temperatures of
Iron - 7 at. pct. Nickel.

Cooling rate °C/sec.	Transformation temp. ± 5 °C.	Cooling rate °C/sec.	Transformation temp. ± 5 °C.
0	730	8,500	458
120	718	10,500	460
200	600	15,000	455
400	604	16,600	442
1,900	537	18,000	413
2,200	508	21,000	414
3,500	463	21,000	410
3,800	463	24,000	412
5,800	460	24,200	411
6,300	460	25,000	412
6,600	460	25,500	413
6,900	460	28,500	410
7,500	460	30,000	410
8,000	460	37,000	412
8,300	458	40,000	413

TABLE VII

Transformation Temperatures of
Iron - 10 at. pct. Nickel.

Cooling rate °C/sec.	Transformation temp. ± 5 °C.	Cooling rate °C/sec.	Transformation temp. ± 5 °C.
0	680	15,300	363
44	585	16,000	360
80	574	17,000	359
150	430	21,500	361
900	410	22,600	364
3,000	378	23,800	360
3,600	366	26,400	364
4,000	365	28,000	362
4,800	365	32,000	362
5,900	364	34,500	363
7,700	360	35,000	363
8,000	360	35,800	357
9,500	360	39,500	362
11,800	365	43,000	358
12,800	365	47,400	361
13,500	365	48,400	362

APPENDIX IV.

SUMMARY OF TRANSFORMATION TEMPERATURES AND CRITICAL COOLING RATES Vs. NICKEL CONTENT.

TABLE VIII

Summary of Transformation Temperatures and Critical Cooling Rates vs. Nickel Content.

Nickel content at. pct.	Massive transformation temp. ± 5 °C.	Martensitic transformation temp. ± 5 °C.	Critical cooling rate °C/sec.
pure iron*	740 ± 10	695	33,000 ± 1,000 °C/sec.
1	675	625	31,500 "
2	625	580	30,500 "
3	580	540	27,500 "
5	485	445	26,000 "
7	460	410	16,500 "
10	---	360	less than 80

*From M.J. Bibby(29)

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